PROCEEDINGS OF THE INSTITUTE

REPORTS OF COUNCIL - OFFICIAL NOTICES

SPECIAL MEETING IN AUSTRIA 1951

A SPECIAL MEETING OF THE IRON AND STEEL INSTITUTE was held in Austria from Thursday, 6th, to Tuesday, 18th September, 1951, by invitation of the Austrian Iron and Steel Institute (Eisenhütte Oesterreich).

The Council of the Institute of Metals invited Members of The Iron and Steel Institute and their Ladies to attend their Annual Autumn Meeting in Italy from 16th to 25th September, 1951. The Council of The Iron and Steel Institute similarly invited Members of the Institute of Metals and their Ladies to attend the Meeting in Austria.

A joint session at which technical papers were presented and discussed was held with the Austrian Iron and Steel Institute in the Stefaniensaal, Graz, on Monday, 10th September. A full programme of visits to works and excursions to places of interest was arranged for Members and Ladies.

RECEPTION COMMITTEE

A RECEPTION COMMITTEE was formed at Leoben to make the arrangements in Austria. Dr. J. Oberegger, President of the Austrian Iron and Steel Institute and General Manager of the Oesterreichisch-Alpine Montangesellschaft, kindly consented to be Chairman. Professor Dr. R. Walzel of the Montanistische Hochschule, Leoben, and Lt.-Col. N. Watson, O.B.E., of Oestereichisch-Alpine Montangesellschaft, acted as Honorary Secretaries. A Ladies Committee was formed under the Chairmanship of Mrs. J. Oberegger.

The Council wish to express their thanks to H.M. Minister in Austria, the Governor of the State of Styria, the Governor of the State of Carinthia, and the Rector of the Montanistische Hochschule, Leoben, for kindly agreeing to be Patrons; to the Council of Eisenhütte Oesterreich and the Chairman and Members of the Reception and Ladies Committees*; to the Council of the Institute of Metals; to the Burgomaster and the City Council of Graz, the Senate of the Montanistische Hochschule, and the managements of the Works* who kindly invited Members and Ladies to visit them; and to the authors of papers and all others who so willingly collaborated in organizing the Meeting.

OPENING SESSION

The Official Opening of the Meeting took place in the Stefaniensaal, Graz, on the morning of Monday, 10th September. Dr. J. Oberegger from the Chair welcomed the Members and Ladies to Austria on behalf of Eisenhütte Oesterreich. He also thanked the Austrian Chancellor and a number of Members of the Federal and Provincial Governments for their presence at the opening ceremony.

Dr. L. Figl, Chancellor, expressed the welcome of the Austrian Government to Members and Ladies from abroad and he hoped that they would find interest and enjoyment during their visit to Austria.

Mr. K. Waldbrunner, Austrian Minister for Industry, expressed the pleasure of the Austrian heavy industries at being able to receive so many important representatives of the British iron and steel industry, and to show them the progress that was being made in the reconstruction and development of the steelworks and engineering industries in Austria.

Professor Dr. F. Platzer, Rector of the Montanistische Hochschule, Leoben, greeted the Members and Ladies in the name of the Faculty of the Hochschule and of all Austrian scientists and technicians.

Mr. R. Mather, President of the Institute, expressed the thanks of all Members and Ladies to those who had so graciously bid them welcome; and to the Chairman, Members, and Honorary Secretaries of the Reception, Executive, and Ladies Committees who were giving so much of their time to making the arrangements for the Meeting. He felt sure that everyone was surprised and deeply affected by the friendliness and warmth of the reception that they encountered everywhere in Austria.

TECHNICAL SESSION

A Joint Technical Session was held with the Austrian Iron and Steel Institute on the afternoon of Monday, 10th September. Mr. R. Mather, President of the Institute, was in the Chair. A paper on "*Investigations*

* The names of those who served on the Reception Committee and a short description of each of the Works visited were given in a Detailed Programme Book issued to each Member taking part in the Meeting.

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into the Effect of Non-Metallic Inclusions on the Hot-Workability of Steel," by F. Rapatz and M. Strobich, was presented and discussed. Thereafter a Lecture on "*Recent Advances in British Open-Hearth Furnaces—A Pictorial Review of Progress in Aerodynamics, Refractories, and Combustion,*" was given by Dr. J. H. Chesters, of The United Steel Companies, Ltd. A short film was shown on "*Fluid Flow.*"

The following papers were specially prepared for the Meeting, but were not presented or discussed:

"The 2000-Year Tradition of the Austrian Iron and Steel Industry," by R. Walzel

"The Present Metallurgical Bases of Austrian Iron and Steel Production," by B. Matuschka.

VISITS AND EXCURSIONS

Gmunden

Approximately 200 Members and Ladies arrived in Gmunden, near Salzburg, in the evening of Thursday, 6th September.

Friday, 7th September

Members were able to visit either Vereinigte Oesterreichische Eisen- und Stahlwerke, A.G., Linz, or Vereinigte Aluminium Werke, A.G., Mattigwerke, and Oesterreichische Metallwerke, A.G., Ranshofen. All those who visited Linz, together with the Ladies, had lunch and spent the afternoon on a steamer on the Danube.

Saturday, 8th September

All Members and Ladies travelled by special train from Gmunden to Graz, stopping *en route* at Trieben; they visited the works of the Veitscher Magnesit, A.G., were entertained to lunch, and saw a film.

Graz

Sunday, 9th September

Members and Ladies had the day free in Graz; a number of Members of Council, and others, were the guests of Mr. R. Rosslyn Penny, H.M. Civil Liaison Officer in Styria, at a cocktail party at which a large number of prominent Austrian Ladies and Gentlemen were present.

A further party of Members and Ladies, who left England on the evening of Saturday, 8th September, arrived at midday on Monday, 10th September.

Monday, 10th September

Members and Ladies attended the Official Opening at the Stefaniensaal in the morning and were the guests of the Austrian Iron and Steel Institute at luncheon in the Schlossberg.

In the afternoon Members took part in the Joint Technical Session with the Austrian Iron and Steel Institute, and the Ladies visited Frohnleiten, Rechberg, and Weizklamm by motor coach.

In the evening Members and Ladies were the guests of Landeshauptmann J. Krainer, Governor of the State of Styria, at a Reception and Buffet given in the Burg, Graz. A short recital of songs was given in the open air by artists of the State Opera.

Tuesday, 11th September

Members travelled by special train to Leoben where they assembled in the Large Hall of the Montanistische Hochschule to receive an official welcome from the Rector and Senate of Hochschule. For lunch they were the guests of Oesterreichisch-Alpine Montangesellschaft in the works hotel at Donawitz. In the afternoon they visited the integrated works at Donawitz and returned to Graz from Leoben by train.

The Ladies made an all-day excursion by bus from Graz to Riegersberg, Bad Gleichenberg, Radkersburg, Mureck, and Leibnitz.

In the evening Members and Ladies were the guests of Dr. E. Speck, Mayor of Graz, for a Reception and Dance in the Kammersaal.

Wednesday, 12th September

All Members and Ladies proceeded by bus and train to visit the Steirische Erzberg (Styrian Ore Mountain) and were the guests of the Oesterreichisch-Alpine Montangesellschaft for luncheon at Eisenerz.

Thursday, 13th September

Members were divided into parties for visits to one or more of the following works:

Gebrüder Böhler, A.G., Kapfenberg
Oesterreichisch-Alpine Montangesellschaft, Kindberg
Felten und Guilleaume, A.G., Bruck-a.-d.-Mur
Schoeller-Bleckmann Stahlwerke, A.G., Hönigsberg-Murzzuschlag
Elin, A.G., für Elektrische Industrie, Weiz
Steyr-Daimler-Puch, A.G., Graz.

Ladies made an all-day excursion by bus to Bruck, Traggös, and the Grüner See.

Pörtschach

Friday, 14th September

Members and Ladies travelled by bus from Graz over the Packstrasse to Pörtschach, stopping for lunch in the Wappensaal of the Landhaus at Klagenfurt. During the afternoon some Members visited Kärntnerische Eisen- und Stahlwerke, A.G., and the Guild of Gun Makers, both at Ferlach.

In the evening Members and Ladies were the guests of the Landeshauptmann F. Wedenig, Governor of the State of Carinthia, at a Reception at the Park Hotel at Pörtschach.

Saturday, 15th September

In the morning Members visited either the Oesterreichisch-Amerikanische Magnesit, A.G., Radenthein, or the Bleiberger Bergswerks Union, Bleihütte Gailitz. The Ladies went by bus to the Ossiacher See. All Members and Ladies assembled for lunch at Radenthein.

Those attending the Institute of Metals Meeting in Italy left by train from Villach in the afternoon; the remainder returned to Pörtschach.

In the evening a Dinner and Dance, at which Mr. R. Mather presided, was held at the Werzer Astoria Hotel, Pörtschach.

Zell-am-See

Sunday, 16th September

Some Members and Ladies returned by train from Pörtschach to London; the remainder travelled by bus over the Grossglockner Mountain Road to Zell-am-See, taking lunch in the Franz Josefshaus overlooking the Pasterze Glacier.

Monday, 17th September

A number of Members visited the Tauernkraftwerke, A.G., at Kaprun.

All Members and Ladies assembled for a Farewell Dinner in the evening and left by train for London at midday on Tuesday, 18th September.

The general feeling of all who took part in the Meeting was one of overwhelming kindness and hospitality in Austria; all were impressed by the great energy and resource being shown by the Austrians in their efforts to rebuild their industries and to restore their country to prosperity.

AUTUMN GENERAL MEETING, 1951

THE AUTUMN MEETING OF THE IRON AND STEEL INSTITUTE was held on Wednesday and Thursday, 21st and 22nd November, 1951, at the Offices of the Institute, 4 Grosvenor Gardens, London, S.W.1. The President, Mr. RICHARD MATHER, occupied the Chair for the greater part of the first day. During his absence his place was taken by Mr. K. HEADLAM-MORLEY, Secretary of the Institute.

Sessions were held from 10.0 A.M. to 1.0 P.M. and from 2.30 P.M. to 4.45 P.M. on the Wednesday, and from 10.0 A.M. to 1.20 P.M. on the Thursday. A buffet luncheon was provided in the Library of the Institute on the Wednesday.

The Minutes of the previous Meeting, held in London on 30th and 31st May and 1st June, 1951, were taken as read and confirmed.

REMARKS BY THE PRESIDENT

The **President**: I am pleased to welcome, on behalf of the Council, all the members and visitors present and particularly Professor H. K. Worner from Australia.

It is my sad duty to report the deaths of ten members since the last meeting, and also of three members whose deaths have not previously been reported. I shall mention only three names. Mr. Fred Clements, who died on 19th September, 1951, was elected an Honorary Vice-President in 1946 and was awarded the Bessemer Medal in 1936. Mr. Sven Gustav Edwin Fornander, of Sweden, one of our best-known overseas members, died on 27th September, 1951, and Mr. Arthur Kipgen, formerly Assistant General Manager of the Arbed Company and Honorary Chairman of the Luxembourg Association of Engineers, died on 11th August, 1951.

The meeting stood in silence for a short time as a token of respect.

The President: We are greatly indebted to the Reception and Executive Committee set up by the Austrian Iron and Steel Institute, and to the Honorary Secretaries, Professor R. Walzel and Lt.-Col. N. Watson, O.B.E., for the organization of the Special Meeting held in Austria from 6th to 18th September, 1951.

You will be pleased to hear that, in accordance with a decision of the Council shortly before the Austrian Meeting, I was able to announce at the main reception in Graz, Austria, that Dr. J. Oberegger, President of the Austrian Iron and Steel Institute and Chairman of the Austrian Reception Committee, had been elected by the Council an Honorary Vice-President of the Institute. We felt that it would be a distinct addition to the quality of the Council to be able to bring him in touch with us in this way.

The state of war with Germany was declared, by Order in Council, to be at an end on 9th July, 1951. The Council resolved, at its meeting shortly after that date, that those of German nationality should now again be eligible for election for membership of the Institute in the ordinary way. It has been mutually agreed with the Verein deutscher Eisenhüttenleute to resume the reciprocal arrangements* which were in force before the war.

The Council has decided to put forward at the next Annual General Meeting the proposal that Captain H. Leighton Davies, C.B.E., should become the next President of this Institute. He joined the Institute in 1918 and will have been a member of the Council for 20 years by the time he takes over the Presidency.

Mr. R. Elsdon, the Librarian of the Institute, is due to retire at the end of 1951 after 47½ years of service with the Institute. I do not think there has ever before been an official who has stayed with the Institute for so long. My own membership goes back over 38 years; I can well remember my early visits to the offices of the Institute, then at 28 Victoria Street, and finding that Mr. Elsdon was the man on whom any ordinary member could depend for friendship and help in any matter whatsoever. (*Applause.*) Very many members have had a similar experience in the course of the last 48 years.

Mr. Elsdon's work has been outstanding—he formed the Library in the first instance, and built it up until we now have a Library which, so far as we can judge, is equal to the best of the libraries of any comparable Institutes. It has been Mr. Elsdon's life work, and I must say that there are many of us who could envy him in being able to look back and say 'There is something which I have done, which is of immense value not only to my contemporaries but also to those who will come afterwards as members of the Institute.'

You will all went to thank Mr. Elsdon for his services and wish him long life and happiness in his retiremen (*Applause*). We hope that we shall continue to see him, and that he will continue to regard this building as a place to which he can come as freely as ever in the past, without the worry of having any particular burden on his shoulders.

The Council has arranged that Mr. A. E. Chattin, the Assistant Secretary, will take charge of the Library in the New Year, and that Mr. A. Post, who joined the staff in 1946, will become Joint Assistant Secretary.

The **Secretary** (Mr. K. Headlam-Morley) reported the receipt of a cordial letter of greeting from the Director of the Spanish Iron and Steel Institute, wishing success to this Autumn Meeting.

CHANGES ON THE COUNCIL

The **Secretary** reported the following changes on the Council since the last General Meeting in May, 1951:

Hon. Vice-Presidents—Professor J. H. Andrew, Sir Arthur Matthews, O.B.E., Dr. J. Oberegger.

Vice-Presidents—Mr. I. F. L. Elliot, Mr. N. H. Rollason.

Members of Council—Mr. H. Boot, Mr. E. T. Judge, Mr. Samuel Thomson.

The following are Hon. Members of Council during their periods of office:

Mr. N. C. Lake, President of the Cleveland Institution of Engineers, in place of Mr. G. B. Thomas.

Mr. F. E. Probyn, President of the Ebbw Vale Metallurgical Society, in place of Mr. J. H. Steele.

Mr. F. K. Neath, President of the Leeds Metallurgical Society, in place of Mr. G. W. Green.

Mr. V. L. Farthing, President of the Liverpool Metallurgical Society, in place of Mr. H. Edwards.

* Details were published in *J. Iron Steel Inst.*, 1951, vol. 169, p. 389.

Mr. L. A. S. Perrett, President of the Swansea and District Metallurgical Society, in place of Captain H. J. Thomas.

Professor R. Hay, President of the West of Scotland Iron and Steel Institute, in place of Mr. W. Barr. (Mr. W. Barr remains a Member of Council in his own right.)

The following are due to retire at the next Annual General Meeting and are eligible for re-election :

Vice-Presidents—Mr. G. H. Latham, Mr. J. Sinclair Kerr, Mr. H. H. Burton.

Member of Council—Dr. J. W. Jenkin, Mr. W. F. Cartwright, Mr. F. Saniter, Mr. T. Jolly, Mr. R. A. Hacking, O.B.E.

ANDREW CARNEGIE SCHOLARSHIPS

The **Secretary** : The following awards have been made since the last General Meeting :

Mr. J. Burke (Liverpool University). £50 (second grant) for work on the decomposition of the carbide constituent in high-purity cast irons.

Mr. M. J. Olney (Cambridge University). £250 for work on the use of a reflecting microscope for high-temperature metallography.

Mr. A. Shelton (Imperial College of Science and Technology, London). £250 for research on the measurement of Poisson's ratio in the elastic range of various metallic materials and also in the plastic state at various degrees of work-hardening.

BALLOT FOR THE ELECTION OF MEMBERS AND ASSOCIATE MEMBERS

Dr. W. N. Hindley and Mr. Raymond Lemmy, who had been appointed scrutineers of the ballot, reported that the following 81 Members and 22 Associate Members had been elected :

Members

Allard, Marc (St. Germain en Laye, France) ; **Aybar,** Manuel (La Felguera, Asturias, Spain) ; **Bartu,** Franz, *Dipl. Ing.* (Carinthia, Austria) ; **Bohomoletz,** Paulo Miguel (São Paulo, Brazil) ; **Broennimann,** Markus, B.S. (La Oroya, Peru) ; **Brown,** Reginald W. H. (Oldbury) ; **Buchanan,** David, B.Sc.(Eng.) (London) ; **Burrows,** Roy F. (London) ; **Campbell,** Michael R., M.A., M.S. (Sydney, Nova Scotia) ; **Cantrell,** John (Manchester); **Carson,** James, O.B.E. (Glasgow) ; **Cattaneo,** Francesco (Milan, Italy) ; **Colepeper,** Leonard G. (Natal, South Africa) ; **Cooper,** Alexander Hyndman, B.Sc., A.R.T.C. (Corby) ; **de Macedo Soares e Silva,** Col. Edmundo, (Rio de Janeiro, Brazil) ; **Dewidels,** Jan Richard, *Dipl. Ing.* (Leeds) ; **Dodd,** John, B.Sc. (Staveley) ; **Doo,** Betty, L.I.M. (Wednesbury, Staffs.) ; **Edmunds,** William T., B.Sc. (Oldbury) ; **Eliasz,** Wladyslaw, M.Sc. (London) ; **Ellis,** Dennis T. (Coventry) ; **Ellis,** Leslie, A.Met., A.I.M. (Sheffield) ; **Everard,** William H. (Sheffield) ; **Fisher,** John C., Sc.D., A.B. (Schenectady, U.S.A.) ; **Fitzgerald,** Timothy (Whetstone, *via* Leicester) ; **Flanigan,** Dr. Alan E. (Los Angeles, California) ; **Frewer,** Reginald A., B.Sc.(Eng.) (London) ; **Gensamer,** Maxwell, D.Sc. (New York, U.S.A.) ; **Granville,** Raymond A., B.Sc. (London) ; **Halm,** Louise (Paris, France) ; **Horsfield,** Alec M., B.A. (Bilston, Staffs.) ; **Hundy,** Bernard B., Ph.D., B.Sc. (Sheffield) ; **Hunt,** John (Sheffield) ; **Hutchinson,** Norman W. (Melbourne, Australia) ; **Icke,** James H. (Rotherham) ; **Intrater,** Norbert, A.M.I.Mech.E. (Haifa, Israel); **Jenkins,** William J., Ph.D., B.Sc. (Glasgow) ; **Kay,** Harry, M.Sc. Tech., M.Inst.F. (London) ; **Knaur,** Fritz, LL.D., *Dipl. Ing.* (Vienna, Austria) ; **Kruse,** Erik A. (Buenos Aires, Argentina) ; **Labrucea,** Antonio (La Felguera, Asturias, Spain) ; **Lawrie,** John A. N. (Granville, N.S.W., Australia) ; **Lewis,** George L. (Bombay, India) ; **Lloyd,** Eos Clwydfryn, B.Sc., L.I.M. (Cardiff) ; **Lloyd,** Eric G., B.Sc.Eng. Met. (Middlesex) ; **MacKenzie,** Archibald G., B.Sc. (Corby) ; **Marshall,** Bernard C., B.Sc. (Natal, South Africa) ; **Martijena,** Armando (Jujuy, Argentina) ; **Martinez,** Giorgio, M.A., A.M.I.Mech.E. (Eastleigh, Hampshire) ; **Marvill,** Ernest (Chesterfield) ; **Matton-Sjöberg** Per E. J., B.Sc. (Domnarfvet, Sweden) ; **Mellars,** Lawrence (Sheffield) ; **Munir,** Mohamed Zaki, Ph.D., B.Sc. (Cairo) ; **Naveira,** Salvador Jose (Buenos Aires, Argentina) ; **Nicklin,** R. J. P. (London) ; **Peretti,** Ettore A. J., D.Sc., M.S. (Notre Dame, Indiana, U.S.A.) ; **Prasad,** Rajendra, B.Sc. (Met.Eng.), B.Sc.(Hons.) (India) ; **Prestrud,** Aksel K. (Oslo, Norway) ; **Quaass,** Stanley T., B.Met.E., M.Eng.Sc. (London) ; **Quijano y Secades,** Javier (Santander, Spain) ; **Rang,** Edmund Jule, M.Sc. (Newcastle-on-Tyne) ; **Reynaud,** Andre (Paris, France) ; **Reynolds,** Thomas (Birmingham) ; **Rodriguez de la Peña,** Arturo (Reinosa, Spain) ; **Ryhagen,** Stig R. (Sandviken, Sweden) ; **Sara,** Edward T., B.Sc. (Sheffield) ; **Sargeant,** Colin W. (West Bromwich) ; **Simons,** Langdon S., A.B. (Paris, France) ; **Singleton,** William, F.I.M. (London) ; **Smith,** Donald Leonard (London) ; **Staples,** R. T. (Birmingham) ; **Stiles,** J. Barrington, J.P., M.I.Mech.E. (Chobham, Surrey) ; **Styren,** Ulf (Oslo, Norway) ; **Unger,** William S. (Düsseldorf) ; **Vargas,** Fernando Gonzalez (Mexico) ; **Waddell,** Thomas K. (Manchester) ; **Warren,** Gordon F. (London) ; **Whale,** Ronald (London) ; **Worley,** Sidney C. (Birmingham) ; **Wuppermann,** Dr. A. Theodor, (Germany) ; **Yapp,** Capt. Anthony Donovan (Sheffield).

Associate Members

Aston, Kenneth (Bilston, Staffs.) ; **Cheetham,** Geoffrey (Sheffield) ; **Coulson,** K. J., B.Sc.(Met.) ; **Gwinnett,** Francis E. (Bilston, Staffs.) ; **Harvey,** Norman B. (Birmingham) ; **Henry,** John C. (London) ; **Hines,** John G., B.A. (Cambridge) ; **Howlett,** Malcolm (Sheffield) ; **Hull,** Derek (Cardiff) ; **Johnson,** Roy H. (Bilston, Staffs.) ; **McNeil,** William (Newcastle-on-Tyne) ; **Martin,** Alan. H. (Bilston, Staffs.) ; **Martin,** Frank L. (Sheffield) ; **Mirza,** Mohammad Razi, B.A.(Hons.) (Southampton) ; **Powell,** Richard (Bilston, Staffs.) ; **Singh,** Govind (Wolverhampton) ; **Smale,** John H. W. (Newcastle-on-Tyne) ; **Thomas,** Idris J. (Llanelly, Carmarthenshire) ; **Tuck,** Charles W. (Cambridge) ; **Walker,** David G., B.Sc. (Melbourne, Australia) ; **Ward,** Robert G. (Cambridge) ; **Whittaker,** Gordon (Bilston, Staffs.).

The **President** declared the candidates duly elected, and announced that the total membership of the Institute was now 4787.

PRESENTATION OF PAPERS

Papers presented for discussion at the meeting dealt with the following subjects :

Wednesday, 21st November, 1951

Morning Session

The Crystal Structure of Iron–Silicon–Carbon Alloys, and

The Metallography of Silicon–Iron Alloys and the Electrical Properties of Silicon–Iron Transformer Sheet.

Afternoon Session

Ingot Moulds.

Thursday, 22nd November, 1951

Morning Session

The Production and Mechanical Properties of Iron and Iron Alloys,

The Physical and Mechanical Properties of Segregates, and

The Structure of Tempered Martensitic Steels.

The full report of the discussion is printed on pp. 221–255, of this issue of the *Journal*.

IRON AND STEEL
INSTITUTE PAPERS

PLANT DESIGN · MANUFACTURE · RESEARCH · DEVELOPMENT

The Structure of Carbides in Alloy Steels

Part II—CARBIDE FORMATION IN HIGH-SPEED STEELS

By H. J. Goldschmidt, M.Sc., F.Inst.P.

SYNOPSIS

An X-ray investigation carried out on filings and electrolytic carbide extracts of high-speed 18–4–1 type steels showed that, apart from the austenite–martensite transformation, a variety of changes in carbide structure can occur on hardening and tempering and with varying alloy content, and these changes are certain to have a considerable influence on properties.

The carbide 'eta' (Fe_4W_2C/Fe_3W_3C) is always predominant, and frequently is the only carbide present; but additional compounds formed are: (*a*) the kappa-carbide (Cr, Fe, W, Mo)$_{23}C_6$, (*b*) an iron–tungsten intermetallic compound ξ', (*c*) a transitory precipitate between η and κ, revealed only after restricted tempering conditions, and (*d*) a carbide V_4C_3 of rock-salt structure, which occurs only if vanadium is present in excess, lesser amounts of this element (and of chromium) entering η and κ.

Internal changes in lattice parameter occur within the eta carbide (owing to differences in alloy content of the carbide) as well as in states of strain and sub-division. The carbide is very hard but, it is suggested, is inherently hardenable, and in this feature is seen a practical application of the carbide as raw material for powder-metallurgical uses.

A new eta-carbide (η^*) of greatly contracted cube-edge has been observed, which is formed with the simultaneous liberation of tungsten.

The partition of alloying elements between matrix and carbide varies with heat-treatment; cobalt, for example, is observed initially (after hardening and short tempering) to reside entirely in austenite and martensite, but, on prolonged tempering, migrates partly into the carbide.

The effect of decarburization on high-speed steels and on pure η is studied, as is the formation of the compound ξ' with which it is associated. The iron–tungsten system has been examined incidentally in this connection, and several structural modifications were observed, differing from the accepted ones.

The nature of high-speed steels is discussed more broadly from the phase-diagram point of view.

A theory is put forward to explain the retention of austenite by an inhibition to decomposition, caused by two competitive carbides tending to precipitate, the two carbides being of different composition but similar free energy, so that the actual nucleation of either carbide is prevented by mutually exclusive conditions of diffusion.

Introduction

THE first part of this investigation, a general survey of carbide systems, which deals primarily with the quaternary system Fe–Cr–W–C, was published earlier.[1] So many interesting features concerning the nature of high-speed steels were found that it was decided to study the carbide formation in these steels with a view to the direct practical application of the results, *e.g.*, in heat-treatment and choice of composition. This paper is mainly concerned with the formation of carbides and intermetallic compounds; the matrix phases (austenite, martensite, ferrite) are referred to only where they form an integral part of the reactions.

Previous investigations on high-speed steel constitutions include the work of Gill[2] and Cohen and Koh,[3] and also Westgren's[4] fundamental studies on the carbide structure. Cohen and Koh concentrated on the austenite transformations and the effect of tempering, while Gill, in examining the carbide segregate, came to the conclusion that this is not materially changed in composition by heat-treatment. In the present paper it is hoped to show that material changes do occur.

The recently published results of Cohen and co-workers[5, 6] and of Krainer[7–9] are in good agreement with the present results in the few instances where they overlap. Reference should be made also to Grossmann and Bain[10] and, for a short summary on high-speed steels, to an article by Monypenny in Smithell's book "Tungsten."[11]

THE POSITION OF HIGH-SPEED STEELS WITHIN THE Fe–Cr–W–C SYSTEM

High-speed steels are best considered within the

Manuscript received 9th June, 1950.

Mr. Goldschmidt is a Research Physicist at the B.S.A. Group Research Centre, Sheffield.

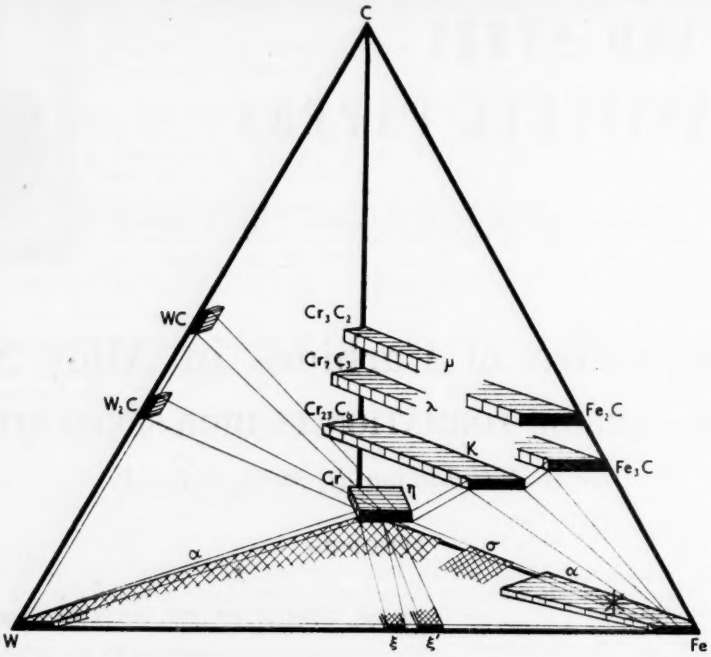


Fig. 1—Schematic space model of quaternary system Fe–Cr–W–C. The asterisk indicates the approximate position of high-speed steels

framework of the quaternary system Fe–Cr–W(Mo)–C; the effect of vanadium will require special consideration.

From the X-ray examination of many commercial alloys and carbide materials included in this system, it has been possible to derive the schematic space model shown in Fig. 1 (for equilibrium at room temperature). The essential carbides occurring are cementite Fe_3C, with approximately 25% solid solubility in chromium; the chromium-base carbides $Cr_{23}C_6$ (κ), Cr_7C_3 (λ), and Cr_3C_2 (μ); the tungsten-base carbides W_2C and WC; and the ternary η carbide Fe_4W_2C/Fe_3W_3C. In κ-carbide, chromium can be entirely replaced by iron and some tungsten to attain the composition $Fe_{21}W_2C_6$ and produce the 'tongue-shaped' single-phase field shown. The η-carbide is the predominant carbide in high-speed steels. The ternary base Fe–Cr–W has not been inserted in Fig. 1, as it has been discussed in a separate paper[12]; the chief phases are the ferrite (α) solid solution and its Cr- and W-rich body-centred cubic isomorphs, the σ-phase FeCr, and two iron tungstites ξ and ξ'. The crystal structures of the various carbides have been discussed in Part I.[1]

In the quaternary model, annealed high-speed 18–4–1 type steels fall into the ferrite $+\eta$ two-phase field; however, very small variations in composition or heat-treatment are capable of introducing or eliminating neighbouring phases, particularly the κ-carbide or the ξ' compound.

COMPOSITION AND CONSTITUTION OF HIGH-SPEED STEELS

The compositions of the series of high-speed steels examined are given in Table I, and in the last column they are expressed in terms of atomic formulae. The 18–4–1 (W–Cr–V) steel, which is the prototype high-speed steel, corresponds atomically with a 6–5–2 composition, illustrating the relative importance of tungsten and chromium. The problem then arises how the elements Fe, W, Cr, V, and C, are partitioned among the constituent phases.

The phase constitutions of steels Nos. 1–11, which were annealed at 620° C. for two days, are ferrite + η-carbide throughout, and from their X-ray patterns (Fig. 8) they might at first sight seem indistinguishable. Differences exist, however, in that:

(i) Traces of a third phase, the κ-carbide, appear in some steels and not in others
(ii) Differences in lattice parameter occur, of both ferrite and η.

The relative amounts of ferrite and η varied only slightly, between limits 80–70% of ferrite and 20–30% of η. The 'traces' of κ present do not exceed 4%, and are generally about 2%. The constitutions and lattice spacings are given in Table II.

An example of the partition of elements between carbide and matrix is given for steel No. 4 (18–4–1) oil-hardened at 1330° C. and tempered 2 hr. at 550° C.:

Phase Constitution	Atomic Formula, on the basis of C = 1
Bulk Steel 70–75% martensite 5–10% austenite 20–25% η-carbide	$Fe_{20\cdot5}W_{1\cdot6}C_{1\cdot2}V_{0\cdot4}C$
Carbide Extract η-carbide, pure	$Fe_{2\cdot9}W_{2\cdot1}Cr_{0\cdot6}V_{0\cdot35}C$*

* Analysis by wt.-%: 27Fe, 63W, 5Cr, 3V, 2C.

This formula for η is in good agreement with the accepted one (Fe_4W_2C) if the chromium and vanadium atoms are assumed to replace iron, not tungsten.

Table I

COMPOSITIONS OF THE HIGH-SPEED STEELS INVESTIGATED

Steel No.	Wt.-%									At.-%								Compositions Expressed as Atomic Formulae on the Basis of C = 1
	C	Cr	W	Mo	V	Co	Mn	Si	Fe	C	Cr	W	Mo	V	Co	Mn	Si	Fe	
1	0·28	3·61	8·68	...	0·28	...	0·31	0·21	86·63	1·35	4·1	2·75	...	0·32	...	0·33	0·44	91·0	$Fe_{7}W_{4}Cr_{2}V_{0.5}C$
2	0·36	3·52	9·50	...	0·40	...	0·42	0·22	85·58	1·76	3·9	3·05	...	0·46	...	0·45	0·46	89·8	$Fe_{11}W_{3}Cr_{2}V_{0.5}C$
3	0·69	4·30	18·8	...	0·90	...	0·27	0·23	74·82	3·59	5·13	6·21	...	1·11	...	0·31	0·50	82·8	$Fe_{21}W_{4}CrV_{0.5}C$
4	0·79	4·08	18·9	...	1·40	...	0·26	0·14	74·42	4·05	4·85	6·32	...	1·72	...	0·31	0·31	82·4	$Fe_{20}W_{4}CrV_{0.5}C$
5	0·73	4·32	18·9	...	1·25	...	0·31	0·23	74·21	3·75	5·10	6·32	...	1·54	...	0·37	0·49	82·4	$Fe_{21}W_{4}CrV_{0.5}C$
6	0·72	4·24	17·6	0·68	0·90	...	0·29	0·23	75·33	3·69	4·98	5·90	0·43	1·11	...	0·31	0·49	83·0	$Fe_{21}W_{3}Mo_{0.5}CrV_{0.5}C$
7	0·73	4·16	18·9	0·53	1·10	1·25	0·45	0·26	72·65	3·81	5·00	6·31	0·31	1·37	1·31	0·50	0·56	81·3	$Fe_{21}Co_{1.5}W_{3}Mo_{0.5}CrV_{0.5}C$
8	0·72	4·64	18·3	...	1·55	5·65	0·31	0·19	68·62	3·75	5·58	6·24	...	1·87	6·00	0·38	0·44	76·2	$Fe_{20}Co_{3}W_{3}CrV_{0.5}C$
9	0·69	4·80	19·2	...	1·50	5·80	0·26	0·14	67·62	3·64	5·75	6·51	...	1·82	6·13	0·31	0·31	75·2	$Fe_{21}Co_{3}W_{3}Cr_{2}V_{0.5}C$
10	0·75	4·78	21·6	...	1·70	18·7	0·34	0·16	51·22	4·04	5·89	7·50	...	2·12	20·3	0·38	0·32	59·3	$Fe_{21}Co_{3}W_{3}Cr_{2}V_{0.5}C$
11	0·72	4·80	22·5	0·65	1·75	15·5	0·30	0·20	53·60	3·87	5·92	7·88	0·45	2·19	17·0	0·39	0·45	62·0	$Fe_{21}Co_{3}W_{3}Mo_{0.5}Cr_{2}V_{0.5}C$

On the other hand, η is known[4] to possess a solubility range from Fe_4W_2C to Fe_3W_3C, and this range is very closely represented by the above empirical result if Cr and V are assumed at one extreme to replace iron completely, giving $(Fe, Cr, V)_4W_2C$, and at the other extreme to replace tungsten, giving $Fe_3(W, Cr, V)_3C$. This ambiguity in positioning is in accordance with the transitional character of Cr and V; atomic sizes would be favourable for Cr and V to replace iron (as they do in ferrite when carbon is absent), whilst valencies and the character of Cr and V as carbide-formers would tend to place them in a carbon environment similar to tungsten. In any case, certain sites in the structure (the 32 *f*-positions, *see* Part I, Fig. 5[1]) are free to be occupied by either Fe or W, and it seems reasonable to assume that Cr and V would fill these sites. The actual composition of the η-carbide will generally be intermediate between A_4B_2C and A_3B_3C; the important point is that, by heat-treatment or variation in gross composition, the effective composition of η can be varied within limits, thus freeing atoms for, or barring them from, entry into austenite or martensite. Although, in the steel assembly, compositions at the iron-rich end of the η field (Fe_4W_2C) would be expected to co-exist with ferrite, this does not necessarily occur. In effect, the portion of the η-field co-existing with ferrite can be made to recede from the α-field, in which case additional phases appear, particularly κ (*see* Table II and p. 192).

Since it is certain that the η-carbide in high-speed steels takes Cr and V into solution, the partition of elements between η and the α/γ matrix can readily be calculated. For a total of 100 atoms the two atomic formulae in the above example are:

Bulk Steel: $Fe_{82\cdot5}W_{6\cdot3}C_{4\cdot8}V_{1\cdot7}C_4$
Carbides: $Fe_{42}W_{30}Cr_{8\cdot5}V_{5\cdot2}C_{14\cdot5}$

The amounts of phases observed from a glancing-angle photograph were approximately: $\alpha + \gamma = 80\%$, $\eta = 20\%$. Thus, out of the 100 steel atoms, 20 enter the carbide (giving $Fe_{3\cdot4}W_{6\cdot0}Cr_{1\cdot7}V_{1\cdot0}C_{2\cdot9}$) and 80 enter the matrix (giving $Fe_{74}W_{0\cdot3}Cr_{3\cdot1}V_{0\cdot7}C_{1\cdot1}$). Tungsten, therefore, enters the carbide almost quantitatively; vanadium favours the carbide by approximately 10 : 7, chromium favours the matrix by 1 : 2, iron favours the matrix by 1 : 9, and carbon favours the carbide by 5 : 2. The relatively high carbon content of the matrix is explained by its martensitic and 10% austenitic (rather than ferritic) character after the tempering treatment (2 hr. at 500° C.).

The carbon liberated by the progressive decomposition of austenite and martensite with temperature enters a new carbide which, as most of the tungsten has already entered η, must contain mainly Cr, Fe, and some V (this is confirmed on p. 195 as κ and *T*).

This formation of a further carbide has an important bearing on the theory of decomposition of retained austenite in high-speed steels.

Impediment to Austenite Breakdown

When austenite decomposes it forms (i) martensite and (ii) η-carbide additional to the η originally present. The reaction, however, proceeds only until most of the tungsten is exhausted, after which another

Table II

HIGH-SPEED STEEL CONSTITUTIONS AND LATTICE DIMENSIONS

Steel No.	Constitution	Lattice Spacing, kX.		Atomic Volumes A, kX.^{3}		ΔA, kX.^{3}
		Ferrite	η	Ferrite	η	
1	α (ferrite) + η (Fe_4W_2C) + trace κ ($Cr, Fe, W)_{23}C_6$	2·8632	$11·067_6$	$11·736_1$	$12·104_4$	0·368
2	$\alpha + \eta$ + trace κ	2·8629	$11·074_9$	$11·732_5$	$12·127_6$	0·395
3	$\alpha + \eta$	2·8638	$11·075_0$	$11·743_5$	$12·127_4$	0·364
4	$\alpha + \eta$	2·8630	$11·075_0$	$11·733_7$	$12·127_6$	0·394
5	$\alpha + \eta$	2·8630	$11·066_5$	$11·733_7$	$12·100_8$	0·367
6	$\alpha + \eta$ + trace κ	2·8615	$11·066_5$	$11·715_3$	$12·100_6$	0·386
7	$\alpha + \eta$	2·8630	$11·067_2$	$11·733_7$	$12·103_1$	0·369
8	$\alpha + \eta$ + trace κ	2·8632	$11·058_4$	$11·736_1$	$12·074_0$	0·338
9	$\alpha + \eta$ + trace κ	2·8640	$11·060_3$	$11·746_0$	$12·080_2$	0·334
10	$\alpha + \eta$ + trace κ	2·8615	$11·062_2$	$11·715_3$	$12·054_4$	0·339
11	$\alpha + \eta$ + trace κ	2·8618	$11·054_7$	$11·719_0$	$12·062_4$	0·343

carbide must form, *i.e.*, κ. The new carbide entails a new configuration of the carbide-former atoms within the austenite lattice. Although an η-like carbon environment is initially established in the parent austenite, rediffusion of the Fe, Cr, and V atoms is necessary to positions from which, after the tungsten responsible for η has been exhausted, they are able to precipitate as κ-carbide. Thus the decomposition of austenite is temporarily inhibited by the necessity for atomic regrouping and for the new carbide to nucleate.

This principle may be likened to one of 'schizophrenia' in alloy transformations; the transformation of a parent phase is inhibited because the free energies of formation of two alternative compounds are similar, and the nucleation of either is prevented by the competitive forming of the other (in this instance, η or κ). It should therefore be possible to remove this impediment to decomposition by adding an element which would facilitate the continued formation of only *one* carbide. Such atoms encouraging η formation are increased amounts of tungsten and molybdenum, and also cobalt (replacing iron) because, if Westgren's suggestion of a carbide Co_4W_3C instead of Co_3W_3C is correct, less tungsten would be required to form an equivalent amount of η. Chromium, even in such small amounts as to enter either η or κ, does not alleviate the impediment.

Although, in moderate amounts, vanadium is capable of entering η and κ, its tendency in excess to form a third carbide, V_4C_3 (*see* p. 201), aggravates the inhibition in carbide precipitation and thus in austenite breakdown. But the inhibition is removed when, beyond a certain content of vanadium and a lower content of niobium, the cubic NaCl carbide formation is assured.

Lattice Dimensions of Ferrite and η

From the spacing results on the annealed steels (Table II) a number of conclusions may be drawn.

(1) *Ferrite*—The ferrite dimensions lie throughout between the values for iron (2·8610 kX.) and chromium (2·8787 kX.), but are relatively near the iron end. The true equilibrium ferrite in high-speed steels is thus of comparatively low alloy content, because all potentially soluble metals (W, Mo, Cr, V) except Co would expand the lattice; these metals are mainly carbide-formers, and this is consistent with the observed high alloy content of the carbides. Cobalt contracts the ferrite lattice, as is shown by the low spacing (2·8615 kX.) for the high-cobalt steels Nos. 10 and 11.

Under decarburizing conditions (p. 198) the ferrite lattice expands considerably, because tungsten is then freed to enter it.

(2) *The η-Carbide*—The η-lattice can dissolve Cr and V, but these elements tend to transfer from η to the κ-carbide when the latter stabilizes. Molybdenum always replaces tungsten in η, whereas cobalt replaces iron.

Structural Anomaly of η—The presence of chromium and vanadium in solution in η causes a slight departure of the structure from cubic symmetry. This anomaly

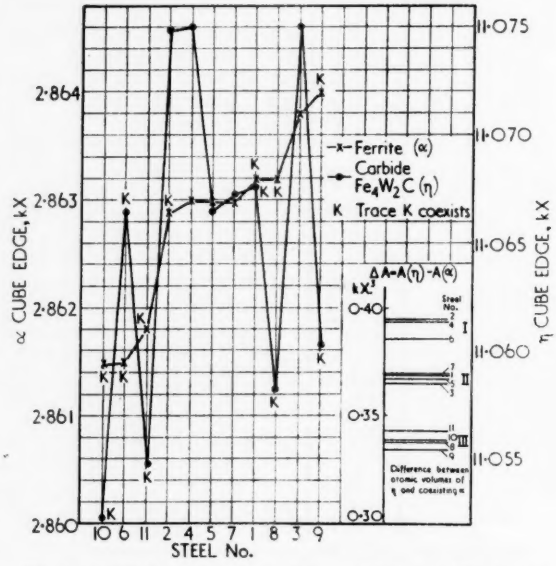


Fig. 2—Lattice parameters for a series of high-speed steels anealed for 2 days at 620° C.

has not, to the author's knowledge, been noted before, and caution is needed when deducing the spacing values; the present figures are based on the (12 2 0) reflection which, with cobalt K_α radiation, appears at large angles adjacent to the ferrite (3 1 0) end-doublet, allowing accurate measurement and comparison with the ferrite parameter. More correctly, η should be termed pseudo-cubic, rather than cubic. X-ray work to elucidate the structure anomaly is proceeding.

(3) *Correlation of Lattice Dimensions with Composition*—The lattice dimensions of both ferrite and η vary appreciably in the series of steels examined (*see* Fig. 2). Since the equilibrium spacing of each phase is a direct function of its alloy content, the apparent lack of a functional relationship between a_α and a_η shows that, for similar heat-treatments, minor alterations in total composition can very considerably alter the balance of elements between matrix and η, the partition of chromium being particularly sensitive.

The results of a graphical analysis in respect of composition are summarized in Table III. Generally a random scatter occurs, but a significant decrease of a_η is observed (Fig. 3) when plotted as a function of Cr, V, and Co contents. This suggests that Cr and V atoms replace tungsten, rather than iron, in the η-lattice; but the small Co-atoms replace iron, and therefore the relative effect of Cr, V, and Co (for these lattice spacings) cannot be observed from these data alone.

(4) *Difference in Atomic Volumes*—The atomic volumes A of α and η (calculated as $\frac{a_\alpha^3}{2}$ and $\frac{a_\eta^3}{112}$) are given in Table II, together with their differences ΔA. There is again a correlation with Cr, V, and Co content (*cf.* Table III); as the amounts of these elements increase, ΔA increases. The values of ΔA are concentrated at or around three distinct levels (*see* inset, Fig. 2):

Group I: $\Delta A = 0{\cdot}390 \pm 0{\cdot}005$ kX.^{3} (steels Nos. 2, 4, 6)
II: $\Delta A = 0{\cdot}367 \pm 0{\cdot}003$ kX.^{3} (steels Nos. 1, 3, 5, 7)
III: $\Delta A = 0{\cdot}338 \pm 0{\cdot}005$ kX.^{3} (steels Nos. 8, 9, 10, 11).

This constancy of ΔA prevails even though the absolute atomic volumes within each group vary widely. Group III contains the higher-cobalt steels; clearly, the atomic volumes of the two phases approach each other as cobalt enters.

In terms of the equilibrium diagram, this grouping reflects the existence of three families of tie-lines within the $\alpha + \eta$ field, each family being substantially parallel within itself, but radiating in three different directions from different portions of the α and η single-phase boundaries. The low-difference group III adjoins the $(\alpha + \eta)/(\alpha + \eta + \kappa)$ boundary, as is shown by the consistent presence of κ in the steels of this group, and by the low absolute dimensions of γ.

Significance of κ-Formation

High-speed steels have generally been considered as essentially iron–tungsten–carbon alloys, with Cr, V, and Mo 'equivalent' to W, assuming that, since these elements are strong carbide-formers, they will also enter η. Grossmann and Bain[10] and Monypenny[11] assumed an approximate equivalence of 1% Cr to 0·5% W, 1% Mo to 1·5% W, and 1% V to 5% W; *i.e.*, they regarded the 18–4–1 steel, for example, as having a '25% tungsten' base. According to the present results this assumption is not altogether justified, because (*a*) κ-carbide appears and (*b*) chromium mainly enters the martensite/ferrite base, and is not wholly a carbide-former.

The occurrence of κ-carbide in high-speed steels has previously been neglected, except for Cohen and Krainer's recent work.[5–9] For example, in the

Table III

RELATION BETWEEN MATRIX AND CARBIDE LATTICE DIMENSIONS AND COMPOSITION

Variant	$a_{ferrite}$	$a_{carbide}$	ΔA
C, %	R.S.*	R.S.	R.S.
W, %	R.S.	R.S.	R.S.
(W + Mo), %	R.S.	R.S.	R.S.
Cr, %	R.S.	C.†	C.
V, %	R.S.	C.U.‡	C.U.
(Cr + V), %	R.S.	C.	R.S.
Fe, %	R.S.	C.U.	R.S.
Co, %	C.U.	C.	C.
(Fe + Co), %	R.S.	R.S.	R.S.
(Fe + Co)/C	R.S.	R.S.	R.S.
(W + Mo)/C	R.S.	R.S.	R.S.
(Fe + Co)/(W + Mo)	R.S.	R.S.	R.S.

* R.S. = Random scatter
† C. = Correlation
‡ C.U. = Correlation uncertain

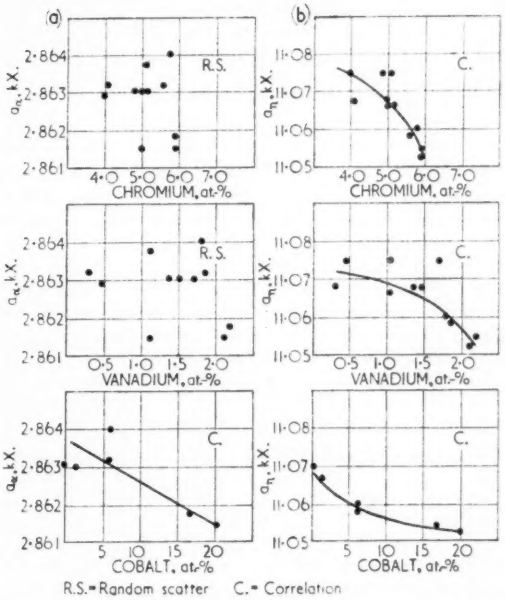


Fig. 3—Variation of lattice parameters of co-existing ferrite and η-carbide with Cr, V, and Co contents: (*a*) Ferrite unit cell; (*b*) carbide unit cell

Table IV

BULK CONSTITUTION OF TEST PIECES BEFORE EXTRACTION

(Steels water-quenched at 1330° C.)

Heat-Treatment	Steel No.	Phases Present			
		Martensite*	Ferrite†	Austenite‡	η
As-quenched	4	↑ Large	...	Approx. 20%	Large
	7	| Large	...	Approx. 5%	Large
	10	| Large	...	1–3 %	Large
Tempered 2 hr. at 550° C.	4	↑ Large	...	Approx. 10%	Large
	7	| Large	...	...	Large
	10	| Large	...	...	Large
Tempered 2 hr. at 600° C.	4	↑ Large	...	Approx. 5%	Large
	7	| Large	...	...	Large
	10	| Large	...	...	Large
Tempered 2 hr. at 700° C.	4	...	Large	...	Large
	7	...	Large	...	Large
	10	...	Large	...	Large
Tempered 2 hr. at 800° C.	4	| Large	...	...	Large
Tempered 2 hr. at 900° C.	4	↓ Large	...	1–3 %	Large

* Martensite tetragonal, but of axial ratio less than 1·005 (*i.e.*, almost cubic). Tetragonal ratio increases in order indicated by arrows
† Only the 700° C. temper produced ferrite, although longer tempering periods at 500° and 600° C. had the same effect
‡ Given as percentage of total (austenite + martensite)

Fe–W–C phase diagrams published by Takeda[13] and Gregg,[14] κ has been omitted and cementite placed in equilibrium with η.

Although only a trace of κ is observed, it has a profound effect on steel properties, especially in incipient precipitation; this is not necessarily detrimental and may enhance the hardness of η, within the lattice of which κ would tend to nucleate. In all the steels in which κ occurs, η possesses low or medium lattice dimensions (*see* Fig. 2), whereas the η-phases of high spacing are *not* accompanied by κ. This indicates that η compositions of low lattice dimensions are saturated in respect of iron and chromium, as might be expected when in equilibrium with κ. On the other hand, there is no apparent relation between the ferrite parameters and the co-existence (or otherwise) of κ; this agrees with the fact that κ precipitates from the η-carbide. In the as-hardened condition, however, the austenite/martensite matrix is a second potential parent constituent for κ precipitation, both *via* secondary η and directly. The fully ferritic samples are naturally soft, and the normal tempering treatment (*i.e.*, several hours at 550° C., instead of 2 days at 620° C.) tends to preserve some austenite, in which case the κ-carbide does not materialize.

The precipitation of κ-carbide proceeds at the same time as the decomposition of retained austenite; both these effects, not only the latter, would contribute to secondary hardening (*see* Cohen and Koh[3]). The formation of κ also partly accounts for the effect of multiple tempering compared with a single temper

Table V

CONSTITUTION OF EXTRACTS AND η LATTICE DIMENSIONS

(Steels water-quenched at 1330° C.)

Heat-Treatment	Steel No.	Phases Present			Lattice Parameter of η, kX.
		η-Carbide	Transitional Phase *T*	κ-Carbide	
As-quenched	4*	Almost pure	...	...	$11{\cdot}066_1$
	7	Pure	...	...	11·076
	10	Pure	...	...	$11{\cdot}069_3$
Tempered 2 hr. at 550° C.	4	Pure	...	...	$11{\cdot}066_0$
	7	Pure	...	...	11·076
	10	Pure	...	...	$11{\cdot}068_2$
Tempered 2 hr. at 600° C.	4	Almost pure	Trace	...	$11{\cdot}065_9$
	7	Almost pure	Trace	...	11·077
	10	Almost pure	Trace	...	$11{\cdot}071_7$
Tempered 2 hr. at 700° C.	4	Almost pure	Trace (increased)	...	$11{\cdot}064_7$
	7	Almost pure†	Trace (increased)	Trace	11·075
	10	Almost pure†	Trace (increased)	Trace	$11{\cdot}070_2$
Tempered 2 hr. at 800° C.	4	Almost pure†	...	Small	$11{\cdot}068_6$
Tempered 2 hr. at 900° C.	4	Almost pure†	...	Small	$11{\cdot}067_4$

* Martensite present in small amounts
† Two η-phases co-exist in these cases, giving (*a*) line-broadening, and (*b*) sharp lines. Phase (*b*) predominates, and is of larger spacing than (*a*)

for an equivalent time, because the intermediate cooling assists (*a*) the transformation austenite → martensite, and (*b*) the nucleation of the κ-carbide, both in η and in martensite and austenite.

EXTRACTED CARBIDE RESIDUES

Although heat-treated powders of the steels have the obvious advantage of giving the total constitution on X-ray analysis, small amounts of additional phases may escape detection. For normal carbides, the visibility limit for the powder method is not more than 1–2%. Therefore, electrolytic extracts were prepared (in 5% HCl solution) and X-rayed. Powder photographs of η-bearing residues are shown in Fig. 9.

The extracts examined were from steels Nos. 4, 7, and 10 (Table I), *i.e.*, 18W–4Cr–1V, 18W–4Cr–1V–1Co, and 21W–5Cr–2V–19Co steels, respectively. The heat-treatments used and the constitutions and η-dimensions obtained are given in Tables IV and V. All the samples were water-quenched after tempering, because the rate of cooling after tempering might affect the structure.

Experimental Results

The main results were as follows :

Chief Carbide—The η-carbide predominates in all the samples, whether they are as-hardened or tempered.

In steel in the hardened condition, η is pure ; it remains pure on tempering for 2 hr. at 500° C., and the fact that the spacing stays constant indicates that no precipitation out of the carbide has yet started.

Secondary Carbides—As the tempering temperature is raised, an additional phase develops in two stages :

(i) An intermediate stage, manifested by a very faint pattern due to a new ' transition phase ' towards κ-formation
(ii) The ultimate stage of κ-carbide formation.

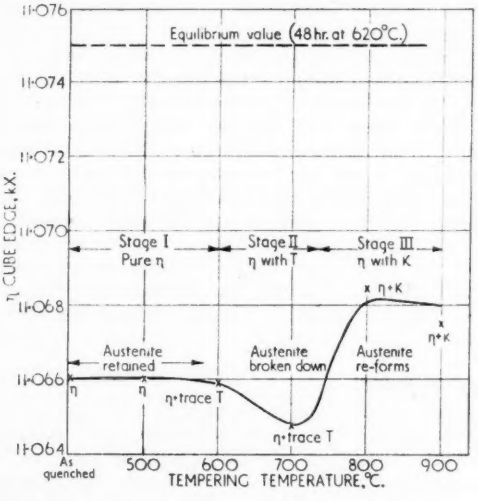


Fig. 4—Lattice parameters of η in extracts from steel No. 4 tempered for 2 hr. at varying temperatures

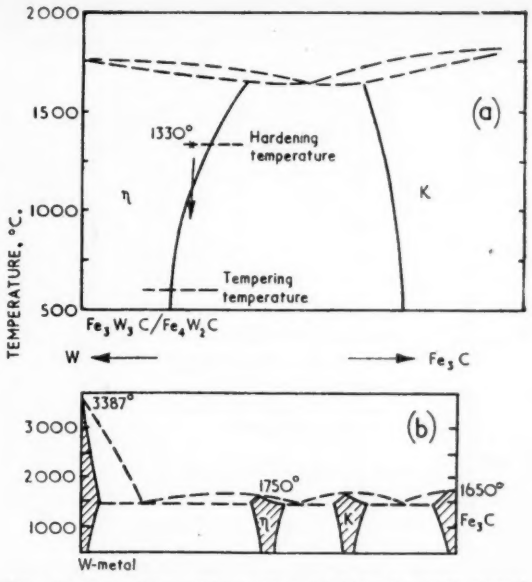


Fig. 5—Transformations of η-carbide in high-speed steels : (*a*) Schematic pseudo-binary section η–κ ; (*b*) section in (*a*) considered as part of series W–η–κ–Fe_3C. (Not to scale. Known melting points are marked, but the exact boundaries are not known)

The formation of κ can be brought about by (*a*) raising the annealing temperature (*e.g.*, to 800° C. for an 18–4–1 steel), or (*b*) prolonging the time at lower temperatures (*e.g.*, the 2-day treatments at 620° C. (*see* p. 190), which in several high-speed steels produced κ in amounts visible even in filings).

The Transition Carbide—The transition carbide *T* is not explained by known phases (such as Fe_3C, Fe_2C, Fe_3W_2, Fe_2W, W_2C, WC, Cr_7C_3, Cr_3C_2, V_4C_3, or their isomorphs), but corresponds to an unstable phase of composition (and probably structure) intermediate between η and κ.

This transition phase begins to appear after tempering at 600° C., but is more evident after 2 hr. at 700° C. ; its formation is probably already initiated at lower temperatures after longer annealing periods. This applies to all three steels.

Lattice Dimensions of η—The formation of a secondary carbide is associated with changes in lattice dimensions of η, as would be expected if η is the parent phase of a κ-precipitate.

The spacings for steel No. 4 are plotted against temperature in Fig. 4 ; the curve reveals three stages, which correlate with the observed presence of austenite in the steels, as follows :

Stage	Carbides (In addition to η)	η-Lattice	Austenite
I	...	Remains constant	Retained
II	*T* forms	Contracts	Breaks down
III	κ forms, plus a *second* η, giving line-broadening	Expands to a higher level than initially; second η is of low spacing	Re-forms

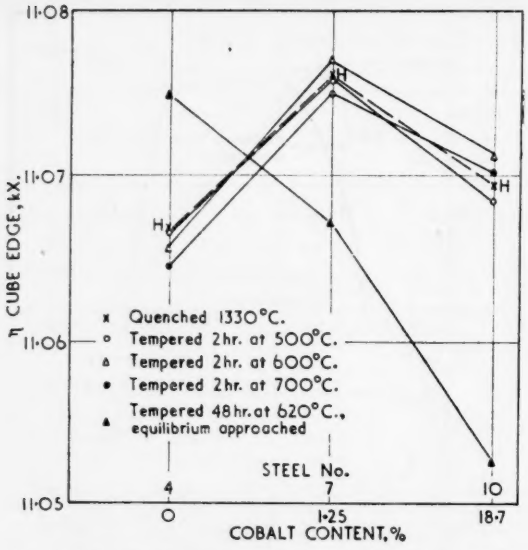


Fig. 6—Lattice parameters of η in residues from high-speed steels of different Co content, as-hardened and tempered at various temperatures

In stage III, the formation of κ and re-formation of austenite on exceeding the A_3 point is associated with an η-expansion, but also with a new phenomenon.

Secondary η-Carbide—The second η-carbide formed shows X-ray line-broadening, in contrast to the still co-existing original η. This may be caused by a high degree of dispersion (particle size of the order of 10^{-6} cm.). Also, the finely dispersed carbide is of considerably smaller mean spacing, *i.e.*, 11·044 kX. compared with 11·063 kX. for normal η. The lesser spacing indicates that the composition of this 'small-particle-size η' is enriched in iron. Its probable origin is as follows :

> The pseudo-binary section W–η–κ–Fe_3C through the quaternary model (Fig. 1) may be represented as in Fig. 5. As the temperature of hardening increases, the iron-rich solution boundary of η extends towards κ until, when hardened from 1330° C., supersaturation in iron occurs. On tempering, finely crystalline secondary η precipitates, which is metastable and represents a preliminary stage to later *T* and κ formation. The sequence of carbides on tempering would thus be :
>
> (i) Normal η
> (ii) Fine-dispersion η
> (iii) Transition structure *T*
> (iv) Final precipitate κ.

Only a small portion of the η-lattice disrupts in this way; the expansion of the bulk carbide in stage III of the curve (Fig. 4) indicates a gain in tungsten, and proceeds in parallel with the formation of the (iron-richer) fine-dispersion η. To a first approximation, the compounds involved correspond to Westgren's solution range Fe_3W_3C to Fe_4W_2C, but within this range the carbon content probably increases with decreasing tungsten content. The η single-phase field in the Fe–W–C system would thus incline along the pseudo-binary section towards κ. The essentially new feature, however, is the retraction of the η-boundary with decreasing temperature. Since η also dissolves some Cr and V (*see* p. 191), a temperature variation in the solubility of these elements would give rise to similar precipitates.

Among the various steels the difference is only quantitative. In steel No. 4, the secondary η appears only after 800° and 900° C. anneals; in steels Nos. 7 and 10 it has already appeared at 700° C., together with the ultimate κ-carbide. Back-reflection photographs show the same effects in the bulk steels, as well as in the carbide extracts.

Practical Example of Different η-Dispersions—Two casts of a 6% W-type magnet steel gave very unequal magnetic performance. X-ray tests showed that, although the matrix in both cases was identical, *i.e.*, pure martensite, the carbide constitution differed considerably: the inferior material contained a single carbide η_A giving sharp reflections, but the superior sample showed *two* carbides, η_A and η_B, of which η_B has the smaller unit cell and gives line-broadening. The carbides η_A and η_B correspond to the two different degrees of dispersion. Knowing this significant difference, practical steps could be taken to guard against it by heat-treatment.

Carbide Strains

An alternative cause of line-broadening is the existence of lattice strain, and in the η-carbide the effects of both particle size and strain-broadening are liable to overlap. It is suggested that the mechanism of this carbide–carbide precipitation is similar to that in the age-hardening of alloys, in which case there is a possibility that the η-carbide can be used as a base raw material of inherent strength and further hardenability. From powder photographs alone it is not possible to differentiate between the relative effects of small particle size, strains, and composition variation, on line-broadening. Indeed, the well-known controversy as to the cause of line-broadening of martensite may apply similarly to the η-carbide, except that the martensite → ferrite transformation is a more sluggish process.

In filings of high-speed steels, which have been water-quenched from a series of decreasing temperatures, η-line-broadening was clearly observed for quenches from the highest temperature (1330°, 1200° C.), but sharp η-lines were evident for quenches from 1130° C. and for tempering (a process analogous to martensite sharpening and ferrite formation). The cooling rate of the filings was far more rapid than that of the bulk samples normally oil-hardened from 1330° C., which (*see* Table IV) had already given sharp η-lines; the line-broadening observed with these filings is probably true line-broadening due to strain.

Effect of Cobalt

Cobalt as an alloying element in high-speed steels is not essentially a carbide-former, but stabilizes austenite, implying increased 'red-hardness' of the resulting martensite. However, some cobalt migrates from matrix to carbide as the time of tempering increases.

In the three steels Nos. 4, 7, and 10 (0, 1·25, 18·7% of Co, respectively), the lattice dimensions of the η-carbide observed in the extracts reflect this feature. Figure 6 shows these dimensions for the different Co contents and tempering conditions. When the 2-hr. 600° C. treatment is considered instead of the 48-hr. treatment, the sequence of γ_i-spacing values changes to :

Steel No. 4 : *Expansion*, from 11·066 kX., by 0·08% (linear)
Steel No. 7 : *Contraction*, from 11·077 kX., by 0·09%
Steel No. 10 : *Contraction*, from 11·072 kX., by 0·18%.

This contraction with time suggests that, as equilibrium is approached, the small cobalt atom migrates to the carbide, particularly as the absolute η dimensions in the Co-richest steel are also much the smallest. To verify this, two extracts (as-hardened, and as-hardened and tempered for 2 hr. at 600° C.) from the 18·7% Co steel were analysed, and *no* cobalt was detected. On the other hand, an η_i-extract from the same steel tempered *for 48 hr.* at 600° C. contained 5·2% of cobalt. (The greater part of the cobalt remains in the matrix phases.) The migration theory for Co was thus confirmed.

The presence of cobalt in the matrix is shown clearly by the ferrite spacings, which decrease from 2·8630 to 2·8615 kX., *i.e.*, by a mean of 0·00011 kX. per 1% of Co added. A comparable mean contraction for pure Fe–Co alloys is 0·00020 kX. per 1% of Co. It is interesting that cobalt possesses a 100% solubility in austenite and considerably less in ferrite, so that while retained austenite and martensite are present the element is tied, whereas on their decomposition it is partly freed to diffuse into η_i.

The variations in η-parameter shown in Fig. 6 are thus proved to be not due to cobalt (which in this case was absent from the carbide) ; they are governed essentially by the W/Fe ratio which, characteristically, increases with increasing spacing. The effects of Cr and V depend on whether they replace W or Fe in their lattice positions.

X-ray examination has shown that cobalt has a pronounced influence on the austenite/martensite transformation (its temperature and rate, axial ratio of martensite, etc.) ; this, however, is a separate subject, and will be published independently.

DECARBURIZATION OF HIGH-SPEED STEELS

The phases (excluding the matrix phases) so far noted in high-speed steels are η, κ, V_4C_3, and the transition carbide *T*. However, if there is a deficiency of carbon (either because of external treatment or because of an unbalanced initial composition), a plain iron–tungstide can be produced.

The Iron–Tungsten System

In the Fe–W binary system two intermetallic compounds have been reported : Fe_3W_2 (ξ) and Fe_2W (ε) observed, for example, by Sykes and Van Horn,[15] whose diagram is reproduced in Fig. 7. The ξ-phase is very stable at all temperatures, and is the chief compound observed in commercial ferrotungsten. Arnfelt[16] carried out an earlier investigation of the system, and determined the structures

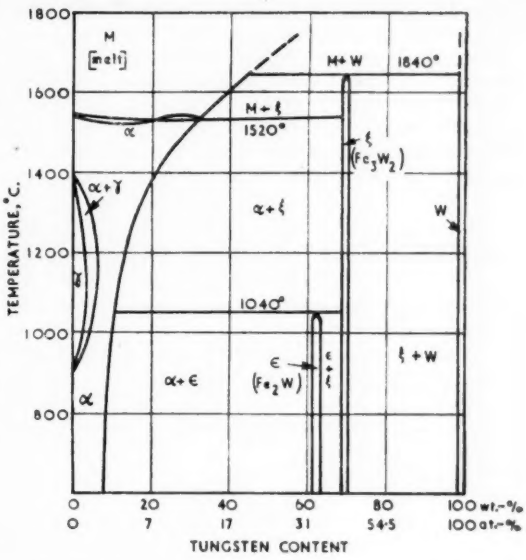


Fig. 7—The iron–tungsten system (according to Sykes and Van Horn[15])

of ξ and ε. His values for the interplanar spacings of ξ in an alloy of composition Fe_3W_2 are compared in Table VI with those observed by the present author. The agreement is good, with the exception of certain lines. The ε-phase (according to Sykes and Van Horn) is stable only up to 1040° C., where it decomposes peritectoidally into ferrite and ξ ; below 1040° C., this phase develops very sluggishly. Some investigators deny the existence of a compound ε, and Arnfelt and Westgren[17] suggest the presence of the phase Fe_7W_6, isomorphous with a cobalt-tungsten compound Co_7W_6.

In the present work, several experiments were carried out to produce the ε-phase by annealing sintered iron–tungsten mixtures ; results are shown in Table VII. At least three distinct structures were observed. The ξ-structures described in Table VII as types I, II, and III, all form modifications of ξ by the omission or re-ordering of tungsten atoms in the lattice, so that a sequence of closely related but distinctive phases is formed. The identification of any of the present types of ξ with Sykes's compound Fe_2W does not appear justified. Slight discrepancies in the interplanar spacings also exist with Arnfelt's compounds (Table VI summarizes all the relevant patterns observed). These results, however, are compatible if they are regarded as representing different stages in successive structural transformations, possibly with Fe_3W_2 and Fe_2W as end-compositions. The further possibility that several allotropic forms of ξ exist cannot be excluded.

Type III is the stable final product of the reaction, at 1000° C., between ξ and pure iron, and the composition of type II agrees with Fe_2W. Since type III will be rather more rich in iron, a formula of about 30 at.-% of W may tentatively be concluded (*i.e.*, approximately Fe_7W_3). This is important, because

type III (denoted ξ' to suggest its close relationship to ξ) is that produced in high-speed steels (*see* below). Figure 10 shows the powder photographs of a decarburized high-speed steel compared with ξ' and with Type II on the verge of transforming to ξ'. Further research on this continuous sequence of transformations in the range 30–40 at.-% of tungsten is being carried out and will be published separately.

The ξ'-Phase in High-Speed Steel

When powder samples of 18–4–1 type steel were held at a high temperature in an imperfect vacuum (pressure 10^{-3} mm. of Hg), the η-carbide was entirely replaced by a phase whose pattern exactly agreed with that of the Fe–W intermetallic compound ξ' (*see* Fig. 10). At the same time, the lattice spacing of ferrite increased considerably, *e.g.*, from 2·8630 kX.

Table VI

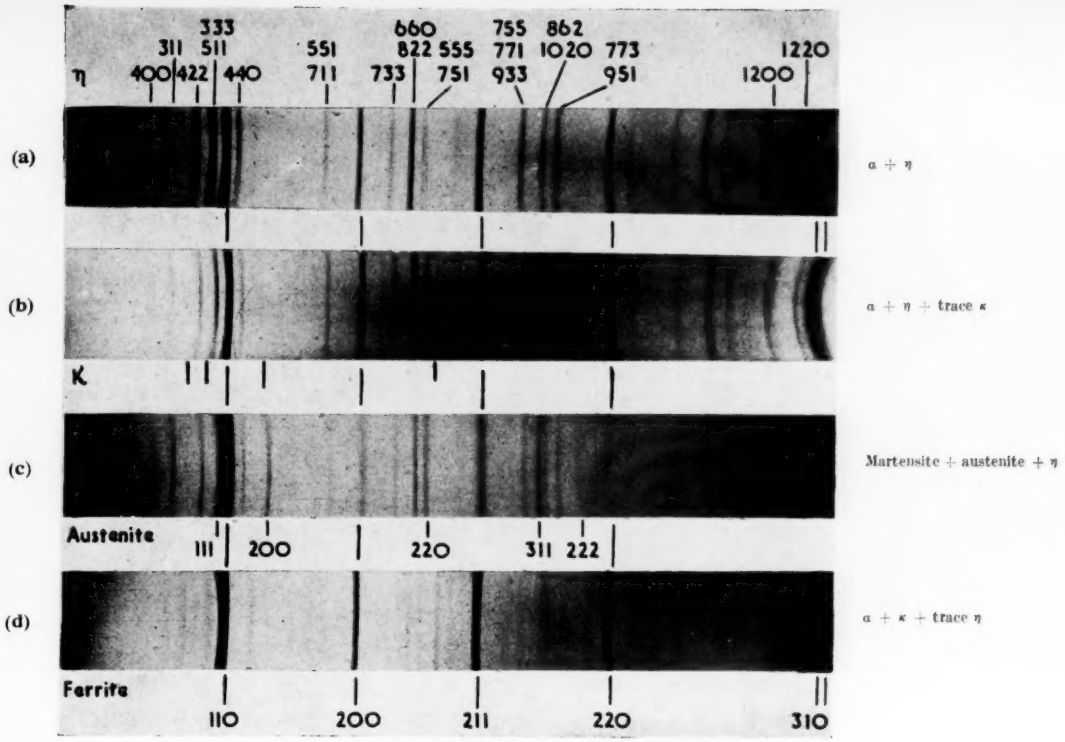
COMPARISON OF INTERPLANAR SPACINGS OF INTERMETALLIC COMPOUNDS IN THE Fe–W SYSTEM

Fe_2W_2 (Arnfelt)	Present Results			Fe_2W (ϵ) (Arnfelt)
	ξ Type I*	ξ Type II†	ξ Type III (ξ')† (Decarburized High-Speed Steel)	
2·76 w‡	2·75 w	...	2·71 vw	2·80 m
2·55 w	2·52 w	...	2·50 vw	...
2·37 s	2·38 s	2·37 s	2·35 mw	2·36 s
2·21 s	2·22 m	...	...	...
2·18 s	2·19 s	2·17 s	2·17 m	...
2·16 s	2·15 w	...	...	...
2·07 s	2·08 s	2·07 ms	...	...
2·05 s	2·05 s	...	2·05 w	2·05 m
2·03 s (F)	...	2·03 ms (F)	2·01 s (F)	2·01 s (F)
1·96 m	1·96 mw	...	1·97 m	1·98 s
1·91 m	1·91 mw	...	...	...
1·82 m	1·82 w	...	...	...
1·79 w	1·79 w	...	...	...
...	1·735 w	...	1·74 w	1·74 m
1·608 s	1·610 m	1·60 m	1·60 m	1·60 m
1·546 w	1·548 w	1·55 w	1·55 vw	1·549 m
...	...	1·510 w	1·515 vw	1·517 m
1·498 w	1·505 w	...	...	...
...	...	1·44 w	1·44 w	1·44 m
1·430 m (F)	1·430 w	1·43 mwd	1·43 s	...
1·423 m	...	...	...	...
1·390 w	1·395 w	1·40 vw	1·40 vw	...
...	1·375 ms	...	...	...
1·369 s	...	1·369 m	1·368 m	1·365 m
1·332 s	1·335 s	...	...	...
1·329 s	...	1·329 ms	1·326 ms	1·326 s
...	1·310 s	1·310 m	...	...
1·302 s	1·302 s	1·305 w	...	...
1·293 s	1·293 s	1·293 w	...	...
...	...	...	1·287 m	1·286 s
1·268 m	1·270 w	1·270 w	...	...
...	...	...	1·233 ms	1·232 s
1·228 s	1·228 s	1·230 s	...	...
1·218 w	...	...	...	...
...	...	...	...	1·201 s
1·185 w	1·186 s	1·187 ms	1·184 m	...
...	...	1·173 w	1·172 s	...
...	1·143 w	1·144 w	...	...
...	...	...	...	1·135 w
...	...	...	...	1·131 w
...	...	1·128 vw	1·130 mw	1·129 w
...	...	1·123 vw	...	1·124 w
...	1·093 s	1·092 s	1·093 m	1·093 w
1·090 w	...	...	...	1·090 m
1·079 w	1·077 w	...	...	...
1·074 w	1·071 w	1·070 vw	...	...
...	1·047 s	...	...	...
1·042 s }	1·041 msd	1·041 s	1·041 s	1·039 s
1·040 w }				
1·026 s	...	1·026 m	1·024 w	1·024 w
1·015 w	1·018 w	1·015 m	1·017 s	1·015 m
...	...	...	...	1·008 m

* ξ Type I = genuine Fe_2W_2

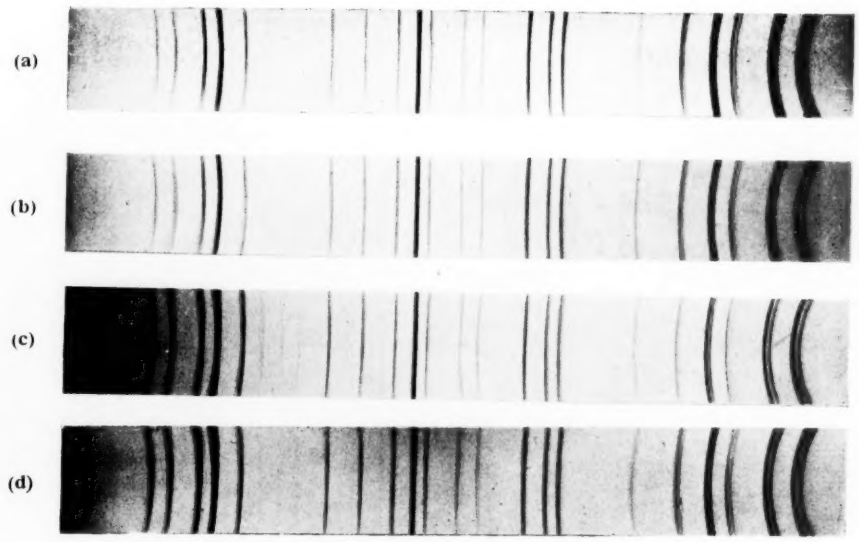
† ξ Type II, ξ Type III (ξ') } = modifications showing different intensities from Type I, and certain line displacements

‡ vw = very weak, w = weak, mw = moderately weak, m = moderate, ms = moderately strong, s = strong ; d = diffuse line, F = ferrite line



(*a*) Steel No. 5. Tempered for 2 days at 620° C.
(*b*) Steel No. 8. Tempered for 2 days at 620° C.
(*c*) Steel No. 4. Oil-hardened at 1330° C., tempered for 2 hr. at 500° C.
(*d*) A κ-bearing steel (1·4% Cr, 2·5% W). Tempered for 2 days at 620° C.

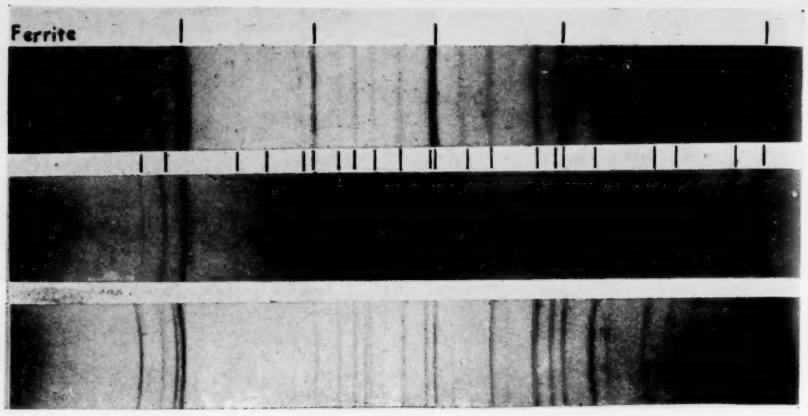
Fig. 8—X-ray powder photographs of high-speed steels



(*a*) As hardened 1330° C.
(*b*) As hardened, tempered 500° C.
(*c*) As hardened, tempered 600° C.
(*d*) As hardened, tempered 700° C.

Fig. 9—X-ray powder photographs of η-bearing residues from high-speed steel No. 10. A subsidiary phase appears in (*c*) and (*d*)

[*Goldschmidt*
[*To face p.* 198

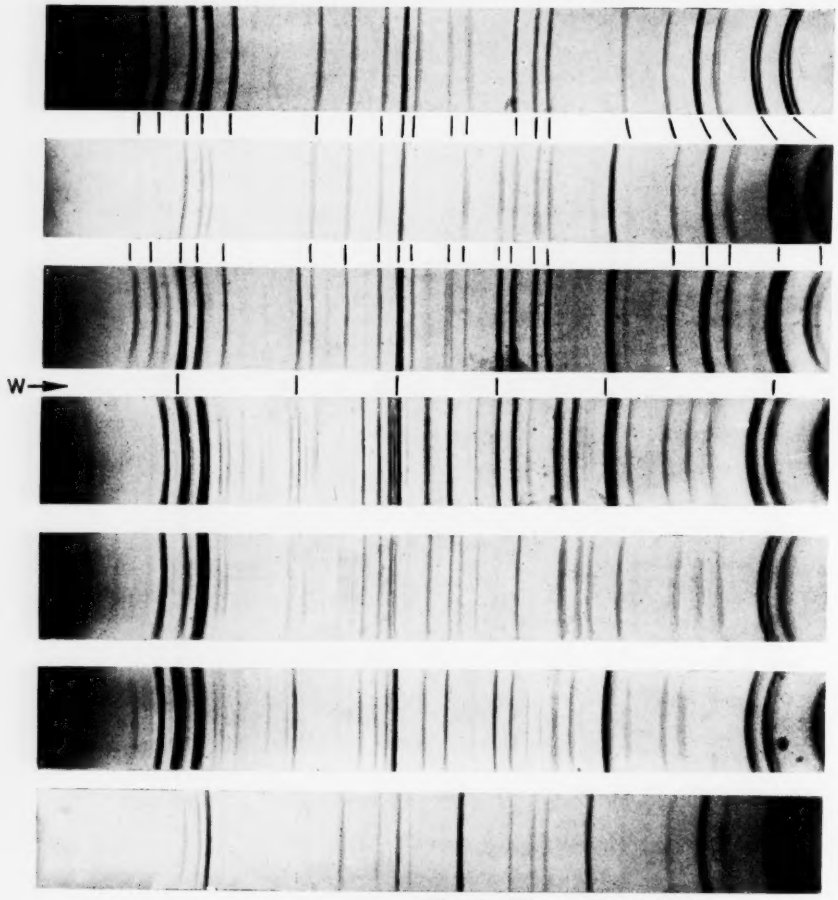


(*a*) Showing ferrite

(*b*) Fe–W binary compound ξ′

(*c*) Fe–W binary compound : transition stage between ξ and ξ′

Fig. 10—X-ray powder photographs of decarburization in high-speed steel No. 4 decarburized at 1250° C. *in vacuo*



(*a*) Pure η

(*b*) $\eta^* + W + \alpha$, produced from $\eta + Fe_3C$ at 900° C.

(*c*) $\eta^* + W +$ trace ξ′, produced from $\eta + \xi$ at 900° C.

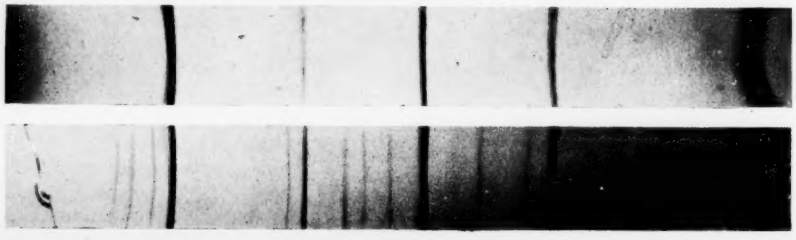
(*d*) $\xi' + \alpha$, produced from $\eta + Fe_3C$ at 1250° C.

(*e*) $\xi +$ trace W, produced from $\eta + \xi$ at 1250° C.

(*f*) $\xi + W$, produced from pure η at 1250° C.

(*g*) $\eta^* + \alpha$, produced from high-speed steel with carbon admixtured, at 1250° C.

Fig. 11—The η and η* carbides



(*a*) Multiple ferrite, produced at 1250° C.

(*b*) Single ferrite + ξ′, produced at 900° C.

Fig. 12—Decarburization of high-speed steel No. 4 + pure Fe powder

Table VII

IRON–TUNGSTEN COMPOUNDS PRODUCED BY INTER-DIFFUSION

Composition, at.-%	Heat-Treatment	Resulting Compound
Fe W		
60 40 (Fe_2W_2)	5 hr. at 1450° C., 24 hr. at 620° C.	ξ Type I*
67 33 (Fe_2W)	5 hr. at 1450° C., 24 hr. at 620° C.	ξ Type I, large α-Fe, small W, small
67 33	16 hr. at 1400° C., 48 hr. at 620° C.	ξ Type I W, small
67 33	16 hr. at 1400° C., 64 hr. at 1000° C.	ξ Type II†
83 17	5 hr. at 1450° C., 24 hr. at 620° C.	ξ Type I α-Fe, small W, trace
83 17	16 hr. at 1400° C., 48 hr. at 620° C.	ξ Type II, but with irregular line widths α-Fe, small
83 17	16 hr. at 1400° C., 64 hr. at 1000° C.	ξ Type III† α-Fe, small

* ξ Type I = genuine Fe_2W_2
† ξ Type II, ξ Type III (ξ') } = modifications showing different intensities from ξ Type I, and certain line displacements.

in the presence of η_i to 2·8697 kX. in the presence of ξ', for equilibrium at 620° C. Similar formation of ξ' occurred under other experimental conditions (*e.g.*, after a hydrogen anneal for 16 hr. at 900° C.). The expansion of ferrite is brought about when a solution of the tungsten is freed by the simultaneous formation of a compound (ξ') richer in iron than the carbide η, according to the equation :

$$\text{Ferrite} + \underset{(\eta)}{Fe_4W_2C} + \tfrac{1}{2}O_2 \rightarrow \text{Ferrite (W-enriched)} + \underset{(\xi')}{Fe_7W_3} + CO\uparrow$$

The fact that tungsten entered ferrite shows that :

(i) Carbide formation is the main cause preventing appreciable solution of tungsten in ferrite, this solution being restored after loss of carbon

(ii) The η-carbide must be more rich in tungsten than is ξ'. This corroborates the suggested composition Fe_7W_3 (30% W) of ξ', instead of the 33·3% W value implied by the η-composition Fe_4W_2C and by Fe_2W (Arnfelt's ϵ).

An approximate estimate of the gain in tungsten (based on a linear extrapolation of the body-centred cube edge a of α-Fe solution to that of W, and substituting W atoms for Fe atoms in α-Fe) can be obtained as follows :

$$\text{Gain} = \frac{a_{\alpha\text{-Fe (decarb.)}} - a_{\alpha\text{-Fe (non-decarb.)}}}{a_W - a_{\alpha\text{-Fe (non-decarb.)}}} \times 100$$

$$= \frac{2{\cdot}870 - 2{\cdot}863}{3{\cdot}158 - 2{\cdot}863} = 2{\cdot}4 \text{ at.-\% of tungsten.}$$

If this 2·4% of W has issued from η of composition Fe_4W_2C, the composition of the ξ' produced must be $Fe_{69}W_{31}$, which agrees fairly well with the formula Fe_7W_3 tentatively concluded (on quite different assumptions) on p. 197.

Further examples of ξ'-production and α-expansion are given in Table VIII for 18–4–1 steel powders, which were hydrogen-annealed for 16 hr. at 900° C. in contact with various admixtures until decarburization occurred. The ξ' phase was formed with nearly all the admixtures. With excess W, as was expected, ξ appeared in addition to ξ' ; it did not appear, however, with Mo. Chromium entered into solution in α *and* in ξ'. The carbon present in cementite and κ had been entirely removed, as had that in η.

Contracted η

The only experiment (Table VIII) in which η_i was preserved was that in which free carbon was present in excess (this is to be expected if decarburization is responsible for ξ' formation). In this case an important new feature was revealed.

The lattice of η_i possesses very considerably smaller dimensions than normal. The new spacing is a = 10·958 kX., compared with the usual value 11·075–11·064 kX. in 18–4–1 steels ; *i.e.*, there is a linear contraction of approximately 1%. The X-ray photographs in Fig. 11 illustrate this effect.

Such a contraction might be caused by :

(i) Loss of tungsten, *i.e.*, enrichment in iron
(ii) Loss of interstitial carbon in η.

Possibility (ii) would imply the existence in the Fe–W–C diagram of a field of η_i homogeneity of appreciable width, extending from Fe_4W_2C/Fe_3W_3C to compositions such as Fe_8W_4C/Fe_6W_6C. However, the evidence is strongly in favour of (i), for the following reasons. Firstly, free graphite coexists with the contracted η_i-phase, and there is still a small amount present after the reaction. Secondly, and more important, this contracted η_i can be produced

Table VIII

DECARBURIZATION OF 18–4–1 STEEL POWDERS WITH VARIOUS ADMIXTURES

(Equal proportions by weight)

Primary Mixture	Product	Ferrite Lattice Parameter, kX.
High-speed steel alone (*i.e.*, $\alpha + \eta$)	$\alpha + \xi'$ throughout	2·8663
,, ,, + Fe		2·8666
,, ,, + 1·2% C steel (*i.e.*, $\alpha + \eta + Fe_3C$)		2·8658
,, ,, + 6% Cr, 1% C steel (*i.e.*, $\alpha + \eta + \kappa$)		2·8659
,, ,, + ferro-tungsten (*i.e.*, $\alpha + \eta + \xi$)		2·8663
,, ,, + Fe–Cr (*i.e.*, $\alpha + \eta + \sigma$)		2·8689
,, ,, + ξ + Fe–Cr (*i.e.*, $\alpha + \eta + \xi + \sigma$)		2·8663
,, ,, + W	$\alpha + \xi + \xi' + W$	2·8663
,, ,, + Mo	$\alpha + \xi' + Mo$	2·8656
,, ,, + C (graphite)	$\alpha + \eta^* + C$	2·8677

Table IX

DECARBURIZATION OF 18–4–1 STEEL AND OF η WITH VARIOUS ADMIXTURES

(Equal proportions by weight)

Primary Mixture	Product	Lattice Parameter, kX.	
		Ferrite	W or Mo
High-speed steel alone	$\alpha + \xi'$	2·8697	
,, ,, + Fe	Three α-phases + trace ξ'	(1) 2·8612 (Fe) (2) 2·8676 (sharp) (3) 2·8676 (diffuse, mean value)	
,, ,, + 1·2% C steel	α + trace ξ'	2·8681	
,, ,, + 6% Cr, 1% C steel	α + trace ξ' (?)	2·8706	
,, ,, + ferro-tungsten (ξ)	$\alpha + \xi' + \xi$	...	
,, ,, + Fe–Cr	$\alpha + \sigma$ + trace ξ'	2·8725	
,, ,, + ξ + Fe–Cr	$\alpha + \xi$ Type II	2·8716	
,, ,, + W	$\alpha + \xi$ + W	2·8637	3·1531
,, ,, + Mo	$\alpha + \xi$ + Mo	2·8631	2·1288
η-carbide alone	ξ + W		
,, + ferro-tungsten	ξ + trace W		
,, + W	ξ + W		
,, + WC	η + WC + W_2C + W		
,, + Fe_3C	$\xi' + \alpha$		
,, + C	ξ Type II + η + W + C		

by hydrogen-annealing† the pure extracted η-carbide alone, the reaction being :

$$\eta \rightarrow \text{contracted } \eta \text{ (predominant)} + W + \text{trace } \xi$$

That is, considerable amounts of *free tungsten* have emanated from η, thus causing the contraction.‡ The η^*-phase was obtained similarly for admixtures of either ξ, W, WC, Fe_3C, or graphite, to pure η, with subsequent annealing.

The η^*-phase can also be produced synthetically from materials not containing η initially. A mixture of ferro-tungsten and carbon gave :

$$\xi + C \rightarrow \eta^* \text{ (large)} + W \text{ (small)} + \text{a residue of } (\xi + C).$$

All these results are consistent with the theory that contraction of η is due to loss of tungsten ; however, the possibility of a subsidiary omission of interstitial carbon atoms is not excluded. In the η unit cell, tungsten and iron atoms randomly occupy the octahedral sites (*cf.* Fig. 5 of Part I[1]), so that the loss of tungsten may be visualized as a simple continuation of the process of replacement of W by Fe, which had already begun during the transition $Fe_3W_3C \rightarrow Fe_4W_2C$.

Under similar decarburizing conditions, therefore, two transformations can be produced :

$$\text{Bulk steel } (= \text{ferrite} + \eta) \rightarrow \alpha + \xi'$$
$$\text{Carbide } \eta \text{ alone} \rightarrow \eta^* + W + \text{some } \xi.$$

The inherent tendency of the ternary η-carbide to liberate tungsten and then contract is, in the presence of ferrite, converted into the actual breakdown of η, the tungsten then being found in the α-lattice and in ξ'. The formation of κ, which is the next richest carbide to η in iron content, is (under decarburizing conditions) replaced by the formation of η^*, the iron content of which may have attained that of κ (Fe : W = 10 : 1, *cf.* 2 : 1 in η).

† Under the same conditions as those used to produce ξ' in the high-speed steel (p. 199).

‡ Contracted η will be denoted η^*.

From a practical viewpoint, the important conclusion is that the high-speed steel carbide, when free, shows tendencies (*i.e.*, the liberation of W and consequent contraction) which must be latent also in the steel, but which are then inhibited by the presence of the matrix.

In 1943, Kislyakova[18] reported the existence of two compounds (η_1 and η_2), isomorphous with Westgren's Fe_4W_2C, in the W–Co–C system. It was thought that these compounds might correspond to the present author's η and η^*, particularly as the lattice dimensions differed in a similar manner :

Kislyakova : $\eta_1 = 11{\cdot}195$ kX., ; $\eta_2 = 10{\cdot}890$ kX.
Goldschmidt : $\eta = 11{\cdot}05$–$11{\cdot}08$ kX. ; $\eta^* = 10{\cdot}958$ kX.

The pattern of η (which is also Westgren's) agrees with η_2, the spacing difference being accounted for by Fe–Co replacement and loss of C. But η^* does *not* agree with η_1 ; the line intensities differ considerably, the parameter of η_1 is larger (not smaller) than that of η_2, and η_1 and η_2 are much richer in tungsten than is η :

Co_2W_4C for η_1
Co_6W_6C for η_2
} Compared with Fe_4W_2C/Fe_3W_3C for η, η^* being richer in Fe, not W, than η.

Also, the deficiency of carbon in η_2 conflicts with the composition of η. However, Kislyakova's compounds are important in that they reveal further possibilities of η replacement and emphasize the general flexibility of the η structure, including its ability to lose carbon without breaking down.

Effect of Temperature of Decarburization

The temperature at which decarburization is carried out has a considerable effect on the results. An example of this is shown in Table IX, which gives results for 18–4–1 steel powders and pure η-carbide, in contact with various admixtures, after treatment for 4 hr. at 1250° C. followed by 24 hr. at 620° C. in an imperfect vacuum (approx. 10^{-3} mm. of Hg).

The η^* phase, so prominent after the 900° C.

treatment (Table VIII), is no longer present, and pure η now gives $\xi + W$; *i.e.*, decarburization has been considerably more effective.

The admixtures reveal several features of fundamental interest.

Pure Fe-Powder Plus High-Speed Steel—Decarburization was complete at both 900° and 1250° C., so that in effect the system is a plain metallic Fe–W–Cr–V system. However, although at 900° C. ξ' is fully developed, at 1250° C. it is still in the matrix solid solution (*cf.* Fig. 12). This ferrite base is not homogeneous, but consists of an aggregate of a ferrite giving line-broadening and another giving perfectly sharp lines. The mean spacing of both the ferrites is identical, agreeing with that of the high-speed steel alone. The ξ'-phase has already nucleated within the parent lattice before precipitation, producing a strained lattice. This phenomenon may be side-band formation, caused by periodic lattice segregates, similar to that observed in permanent magnet alloys.[19] The alloy will probably show age-hardening effects.

These results might be explained from the Fe–Cr–W phase-diagram investigated by the author,[12] where, in the α single-phase field, a miscibility gap opens out with decreasing temperature, and ξ and ξ' precipitate. Iron in excess of that present in the high-speed steel composition is required to produce this effect, and the composition is thus nearer that of the primary α-field and facilitates solid solution at high temperatures. Tempering at 620° C. following treatment at 1250° C. was only sufficient to initiate precipitation, whereas after treatment at 900° C. complete carbide precipitation was possible.

The observations on the high-speed steel with carbon and other admixtures conform to the same principle: at the higher temperature, 1250° C., ξ' is in solution but there is incipient precipitation.

Tungsten Addition—The lattice spacings of the α-matrix (Table IX) show an appreciable tungsten solid solution throughout, *except* where free W or Mo was added to the high-speed steel (2·8637 kX. with free W, 2·8697 kX. without free W). This means that the added tungsten provides a matrix at which the tungsten liberated from the decarburized η (which otherwise would have entered the ferrite solution) can nucleate; the free W or Mo powders possess a 'seeding effect,' accelerating the precipitation from the supersaturated solution in ferrite.

Ferro-Alloy Additions—The ferro-tungsten and ferro-chrome additions (Table IX) show that it is possible to produce from decarburized high-speed steel the ξ, ξ', ξ Type II, and σ phases, in a perfectly systematic manner, in accordance with the Fe–Cr–W phase diagram.[12] Also, the ξ Type II here appears as a stage preparatory to σ-formation, as Cr replaces Fe. The sequence of decarburizing reactions is thus:

(i) Pure high-speed steel gives ξ'
(ii) Addition of ferro-tungsten gives $\xi' + \xi$
(iii) Addition of ferro-chrome and ferro-tungsten gives ξ Type II
(iv) Addition of ferro-chrome gives σ.

Decarburization of η-Carbide Extracts—The decarburization of pure η and of η with various admixtures (Table IX) produced ξ and free W, except when carbon and carbides were added. Cementite alone, when treated under the same conditions, was decarburized to pure α-iron; when treated together with η, it was decarburized to α-Fe + ξ. Tungsten carbide treated with η enhanced decarburization resistance to some extent. The formation of ξ Type II in the presence of free graphite seems anomalous, but decarburization is still possible, despite the presence of excess carbon, if any one η-grain decomposes at a higher rate than the rate of diffusion into it of the surrounding carbon. To prevent decarburization, there should be speedier carbon diffusion for high temperatures and finer particle size.

Synthesis of η by Carburizing—So far, only the reaction $\eta \rightarrow \xi$ (ξ') has been demonstrated; however, by experimentally carburizing ferro-tungsten at 950° C. in graphite, it has been possible to reverse this transition and also to produce the η-carbide from ξ.

SOME SUPPLEMENTARY RESULTS

The constitution η-carbide + matrix is characteristic of all high-speed steels—in fact, these steels might be defined as those in which η is the only, or predominating, carbide. Examples of other constitutions which the author has investigated may be of interest.

Stability of η in Liquid and Solid

To show that, even at the highest temperatures, the η-carbide is stable and does not enter into solid solution in the austenite matrix, samples of a high-speed steel were water-quenched out of the liquid, from 1330° and 1250° C. In all cases the amount of η was not appreciably less than in the annealed alloy. Although this does not prove conclusively that η *did* exist in the free state at the liquid and high solid temperatures—this can be determined only by means of a high-temperature X-ray camera—there would otherwise be instantaneous formation of η from austenite on quenching, no diffusion being involved. Comparing the austenite and the complex η unit cells, such spontaneous carbide formation seems most unlikely, and the existence of free η at all solid temperatures, and of molecular aggregates of η-like configuration in the liquid, is highly probable.

The complete stability of η at all solid temperatures has been observed in 18–4–1 steels after numerous types of heat-treatment, except where decarburization occurs. These results, which form part of an investigation on martensitic structures, fall outside the scope of the present paper. However, the lattice-strain effect (line-broadening and sharpening) in the η-carbide is a major factor, which should be emphasized in any discussion on the structure of high-speed steels.

A Vanadium-Rich Steel

An annealed high-speed steel containing 4·5% of vanadium* revealed three phases: ferrite, η, and V_4C_3 (NaCl-type) of spacing $a_0 = 4{\cdot}17$ kX. The

* Composition: C, 1·25%; W, 14%; Cr, 4·3%; V, 4·5%; Mo, 0·2%.

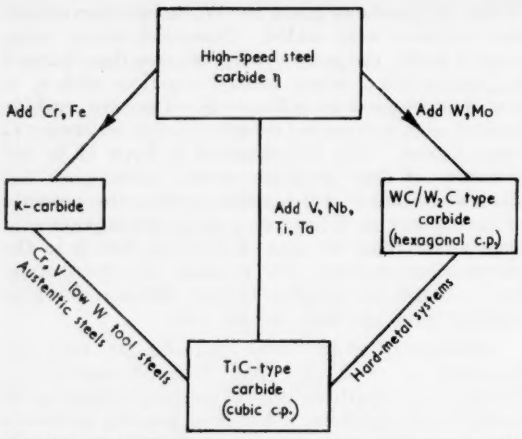


Fig. 13—Classification of types of tool materials according to the structure of carbides contained in them

appearance of the vanadium carbide indicates that :

(i) The amount of V soluble in η is limited. The saturation boundary is at some point between 1·75% V (steel No. 11, Table I) and 4·5% V, and, judging from the line intensity, is probably near 2% V

(ii) The NaCl type of carbide exists in direct equilibrium with η. This is important, because the structure is produced by Ti and Nb as well as by V. Niobium carbide has also been found to co-exist with η in heat-resisting alloy steels. Much lower atomic percentages of Nb have a similar effect to V in high-speed steels, and it appears that vanadium is not only a carbide stabilizer but also renders η precipitation-hardenable, by altering its composition above the retracting phase boundary $\eta/\eta + V_4C_3$. There is therefore a basic functional difference between the behaviour of vanadium and chromium in high-speed steels, since each tends ultimately to precipitate a different carbide[5,6]

(iii) From the point of view of structure systematics, this scheme fits perfectly into the principles outlined in Part I.[1] There is a triangular relationship between the three cubic phases : with η as a basis, either κ or the cubic close-packed structure can form, stimulated respectively by Cr and Fe, and by V, Nb, Ti, and Ta. In addition, W and Mo, in excess, produce the hexagonal close-packed WC/W_2C (Mo_2C). The relationship between these carbides is shown in Fig. 13.

This scheme systematizes tool materials of very different varieties, representing the hard-metal TiC–WC system of cemented carbides and the lower alloyed chromium and vanadium types of steels, with high-speed steels occupying a central position.

η-Formation from Hard Metal

When tungsten-carbide type hard metal was melted with Armco iron, the following phase changes were revealed :

$$\underbrace{WC + TiC\ (small) + Co}_{(Hard\ metal)} + Fe \rightarrow Ferrite + \eta + \kappa.$$

This result gives, in effect, a cross-section of the Fe–W–C diagram, and confirms that (*a*) Fe combines with WC to form η ; (*b*) η, κ, and α-Fe co-exist, the alloy occupying a κ-richer part of the $\alpha + \eta + \kappa$ region than do the high-speed steels ; (*c*) Ti and Co enter these three matrices. Titanium enters η and κ, and therefore, like vanadium, must exceed a certain amount before free TiC is established.

Molybdenum in High-Speed Steel

An 18–4–1 type steel containing 1·3% of Mo and 11% of W, did not differ in constitution from normal 18–4–1 steel, in either the as-hardened or the as-tempered conditions. Molybdenum replaces tungsten in the η-carbide, but with high Mo-content there is a tendency to form σ-phase on loss of carbon.[12]

2% and 6% Tungsten Steels

The fact that as the tungsten content of 18–4–1 steel is reduced the κ-carbide increases at the expense of η, was confirmed by a 2% W type tool steel. These steels represent the κ-rich side of the $\alpha + \kappa + \eta$ region, whereas 18–4–1 steel represents the η-rich side.

'Spoiling' owing to WC formation in tungsten-magnet steels[20] was observed in several cases. For example, with a magnet steel of the 6% W type, there was a considerable difference between the magnetic energy values of two samples of the same steel cast, the constitution of the carbide residues being :

High BH_{max} : $Fe_3C + \kappa$
Low BH_{max} : WC + η.

The probable explanation is segregation of tungsten such that, in effect, the $Fe_3C + \kappa$ and $\eta + WC$ phase fields are represented, instead of the $\eta + \kappa$ phase field which this steel normally occupies.

Phase-Diagram Relationships

Within the framework of the Fe–Cr–W–C diagram (Fig. 1), high-speed steels occupy the $\alpha + \eta$ field,

Table X

EQUILIBRIUM PHASES SURROUNDING η FOR EXCESS OR DEFICIENCY OF VARIOUS ELEMENTS

Element in Excess	Element in Deficiency	Phase formed (in addition to η)
Fe	...	Ferrite
Fe and C	...	κ
C	...	Graphite
W and C	...	WC or W_2C
W	...	Free W
W	C	ξ and free W
...	C	ξ and ξ' (ξ' forms sluggishly below approx. 1000° C.)
Fe	C	Ferrite and ξ'
Cr	C	Ferrite and ξ
Cr and C	...	κ
Cr and W	C	Primary Cr–W solid solution
W, Cr, and C	...	W_2C, WC, and λ (for different C : W : Cr ratios)
V	...	Cubic V_4C_3
Co	...	Replaces Fe in η and in ferrite
Mo and C	...	Mo_2C and Mo replacing W in η
Mo	C	ξ and free Mo

but the addition or subtraction of alloying elements promotes a tendency to form small amounts of either κ-carbide, the NaCl type of carbide (*e.g.*, V_4C_3), the ξ or ξ' phase, or free tungsten. Each alloying element in high-speed steel enhances or retards one such tendency. Moreover, elements can distort or contract the actual η lattice (*e.g.*, by the formation of a suspended solid solution or η^*), in addition to exerting an influence on the austenite/martensite transformation.

The known phases surrounding η on all sides and forming low-temperature equilibria with it are :

Ferrite/ξ'/ξ/W/W_2C/WC/C (graphite)/κ/NaCl-family
(Mo)(Mo_2C) (V_4C_3, TiC, Nb_4C_3, TaC).

The possibility that further equilibrium phases exist cannot be excluded. The effects of excess of (or deficiency in) certain elements are given in Table X.

On the C-rich side, the result that η is in direct equilibrium with graphite answered a fundamental question in the construction of the Fe–W–C ternary system. There were two alternative and mutually exclusive equilibria :

(i) $\left\{\begin{matrix}\kappa \\ Fe_3C\end{matrix}\right\} \longleftrightarrow \left\{\begin{matrix}WC \\ W_2C\end{matrix}\right\}$

(ii) $\eta \longleftrightarrow C$,

and since (ii) applies, the κ or cementite phases cannot be in direct equilibrium with WC or W_2C.

On the W-rich side, certain observations suggest that η and WC do not form a duplex region at all temperatures. After heat-treatments of 18–4–1 steel with WC, a new pattern was observed, which might correspond to a new compound, not yet defined, forming peritectoidally from the $\eta \longleftrightarrow$ WC and $\eta \longleftrightarrow W_2C$ equilibria prevailing at high temperatures. This question is being investigated.

RECENT WORK BY COHEN AND KRAINER

Blickwede, Cohen, and Roberts[5,6] and Krainer[7–9] have recently published papers on high-speed steel constitutions. Their work overlaps only slightly that of the author's, but where comparison is possible the results are in very good agreement.

Cohen and his co-workers investigated the effect, on a 6% W, 5% Mo, 4% Cr base steel, of increasing V and C contents. The compounds M_6C, $M_{23}C_6$, M_3R_2, and MC which they observed agree with the present author's η, κ, ξ', and V_4C_3 phases, respectively, but Cohen does not distinguish between the present different ξ-types, and accepts the 3 : 2 ratio for Fe : W. The phase assemblies they observed also conform, except that κ and V_4C_3 are predominant, whereas in the present work they are only subsidiary to η. Cohen also states that the κ-carbide goes into solution on austenizing (*cf.* the present observation that it precipitates on tempering), stabilizing the austenite and serving as a carbon reservoir. Both Cohen and the present author have confirmed that κ-carbide prevents the formation of the brittle M_3R_2.

Krainer[7] examined high-speed steel systems by X-rays, varying the compositions by pairing any two of the metals W, Mo, Cr, V, Ti, and (Ta–Nb), and recording the spacing variations of the carbides formed. These carbides agreed with the present η, κ, and NaCl-type carbides. His conclusions that most of the W and Mo is present in the η-carbide and that Cr is partitioned between α and η, agree with the present author's, as does the observed tendency of the η-parameter to decrease with increasing Cr and V contents. Krainer also notes the sensitive effect of heat-treatment on partitioning of elements and lattice dimensions (*cf.* the suspended precipitations discussed on p. 196). However, his statement that excess carbon appears as cementite remains unexplained, since κ ought to be between η and Fe_3C, unless κ-carbide tends to decompose.

Krainer's second paper[8] discusses the carbides $Fe_{21}W_2C_6$ and $Fe_{21}Mo_2C_6$ (the present κ) and their equilibria with η and ferrite, and investigates the Fe–W–C and Fe–Mo–C diagrams on a wider composition basis. An appreciable η-homogeneous range can co-exist with ferrite (a point on which the author and Krainer agree), but Krainer's conclusion that the η-ranges with Fe/W ratios of less than 2 : 1 do not correspond to equilibrium, is not thought justified.

CONCLUSIONS

(1) The high-speed steel carbide, although very stable, is by no means immutable, as had previously been believed. In addition to the matrix transformations, high-speed steels show changes in their carbide constitutions, and the practical effect of this has to be considered.

(2) The η-carbide, Fe_4W_2C/Fe_3W_3C, is always the predominant and often the exclusive one in 18–4–1 type steels. Variations in composition and heat-treatment, however, can produce three types of changes : (*a*) in the η lattice dimensions, *i.e.*, substitution of Fe and W for each other and by other elements ; (*b*) in the state of internal strain and dispersion of η ; and (*c*) in the formation of compounds additional to η.

(3) For low-temperature equilibrium, at certain compositions ferrite and η are accompanied by traces of the κ-carbide $(Cr, Fe, W, Mo)_{23}C_6$.

(4) In phase-diagram representation (Fig. 1), high-speed steels are located in the $\alpha + \eta$ two-phase field, but tend to enter either the $\alpha + \eta + \kappa$ or the $\alpha + \eta + \xi'$ three-phase fields, according to slight variations in composition or heat-treatment. Cementite is entirely excluded from equilibrium.

(5) The lattice dimensions of ferrite and η vary appreciably in the various steels, indicating that the partition of elements between matrix and carbide is very sensitive to slight variations in gross composition.

In 18–4–1 steel, for example, tungsten joins the carbide almost quantitatively, vanadium favours the carbide by 10 : 7, chromium favours ferrite by 1 : 2, and iron favours ferrite by 1 : 9. Carbon, all of which eventually joins the carbide, is present in the matrix in appreciable amounts even after tempering, so long as some austenite is retained.

Cobalt joins the matrix after hardening, but with increasing tempering time it partially migrates into the carbide, to attain a definite partition between the two phases. Both Cr and V can be partially dissolved in the η-lattice. The formulae in the range Fe_4W_2C to Fe_3W_3C can be realized by assuming that Cr and V replace alternatively Fe or W. Molybdenum is equivalent to tungsten.

A survey of the lattice dimensions of ferrite and η indicates a correlation with Cr, V, and Co content. Differences in the atomic volumes of α and η fall into three distinct groupings, corresponding to three families of tie-lines.

(6) Ferrite in true equilibrium in high-speed steel contains relatively small amounts of alloying elements, which, except for Co and Cr, mainly lodge in the carbide.

(7) Although κ-carbide formation is enhanced by both chromium and vanadium, the latter, in excess, differs fundamentally from Cr, in its tendency to form an NaCl-type carbide (V_4C_3).

(8) The retention of austenite is believed to be due to an impediment to its breakdown, caused by the competitive effect of different carbides of similar free energy tending to precipitate, so that the nucleation of any one is inhibited. The competitive carbides are η, κ, and (for V, Ti, or Nb additions) the NaCl carbide. One method of accelerating the decomposition is to move the equilibrium in definite favour of *one* of the alternative precipitants.

This principle of a transformation being frustrated by several secondary compounds of similar free energy attempting to form may be of wider metallurgical use.

(9) The κ-phase is capable of precipitating out of the main η-carbide. This internal 'carbide–carbide' precipitation produces lattice strains and probable hardening effects analogous to those in martensite. Simultaneous occurrence of 'primary' and 'secondary' η phases is evidenced by samples showing both sharp and diffuse patterns in parallel.

(10) The precipitation of κ proceeds *via* a transition structure T' observed in carbide extracts at higher ranges of tempering temperatures (for a given time).

(11) A correlation (*cf.* Fig. 4) exists between (*a*) the carbide formed, (*b*) the presence or absence of austenite, and (*c*) the η lattice dimensions. There is a further intermediate stage, that of η in a highly dispersed and/or strained state; the complete sequence of carbide stages is therefore :

Supersaturated η/secondary η/transition structure T'/final κ.

(12) The extracted η-carbide may be of considerable interest as a raw material for sintered hard metals.

(13) Decarburized high-speed steel contains the compound ξ' (approximately Fe_7W_3) observed in the binary system Fe–W. Decarburization is accompanied by an expansion of the ferrite lattice, consistent with ferrite dissolving the tungsten liberated from the former η-carbide. (Decarburization of the stable η-carbide should not occur for normal industrial heat-treatments or in normal tool usage; however, in metallurgical investigations of high-speed steel failures, attention should be paid to the possible incipient local formation of this brittle ξ' compound.)

(14) The Fe–W binary system (studied independently) reveals two structurally related phases ξ and ξ', with intermediate transition stages. There appears to exist a series of compounds, of between 30 and 40 at.-% of tungsten, which is of considerable theoretical interest, the ξ-phase and the σ-phase presenting associated problems.

(15) The high-speed steel carbide η can, by heat-treatment, be transformed into an isomorph (η^*) of much smaller cube edge (*i.e.* 10·96 kX. compared with 11·07 kX.), free tungsten being produced simultaneously. It is believed that this isomorph has not been noted before. It can be produced also by carburizing ferro-tungsten. The tendency of free η to form η^* and W is important, as it must be latent also in the steel (where, however, it is inhibited and replaced by ξ' formation).

(16) The solubility of vanadium in the η-carbide is limited to 2–3%; titanium and niobium behave similarly to vanadium in producing the NaCl-type carbide (although lower atomic percentages of Nb are required), and excess tungsten and molybdenum encourage the hexagonal close-packed carbides. Tool materials can be systematized on this basis (Fig. 13).

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References

1. H. J. GOLDSCHMIDT : *J. Iron Steel Inst.*, 1948, vol. 160, pp. 345–362.
2. J. P. GILL : *Trans. Amer. Soc. Met.*, 1936, vol. 24, p. 774.
3. M. COHEN and P. K. KOH : *Ibid.*, 1939, vol. 27, pp. 1015–1046.
4. A. WESTGREN : *Jernkontorets Ann.*, 1933, vol. 88, pp. 1–14.
5. D. J. BLICKWEDE, M. COHEN, and G. A. ROBERTS : *Trans. Amer. Soc. Met.*, 1950, vol. 42, pp. 1161–1189.
6. D. J. BLICKWEDE and M. COHEN : *J. Met.*, 1949, Sept., pp. 578–584.
7. H. KRAINER : *Arch. Eisenhüttenw.*, 1950, vol. 21, pp. 33–38.
8. H. KRAINER : *Ibid.*, pp. 39–41.
9. H. KRAINER and R. MITSCHE : *Ibid.*, 1949, vol. 20, pp. 197–198.
10. M. A. GROSSMAN and E. C. BAIN : "High Speed Steel." New York, 1931 : John Wiley and Sons, Inc.
11. C. J. SMITHELLS : "Tungsten." London, 1945 : Chapman and Hall, Ltd.
12. H. J. GOLDSCHMIDT : Symposium on High Temperature Steel and Alloys for Gas Turbines, pp. 249–257. *Iron Steel Inst., Spec. Rep. No.* 43.
13. S. TAKEDA : *Technology Reports of the Tôhoku Imperial University, Sendai.* 1930, vol. 9, pp. 627–664; 1931, vol. 10, pp. 42–92.
14. J. L. GREGG : "The Alloys of Iron and Tungsten." New York and London, 1934 : McGraw-Hill Book Co., Inc.
15. W. P. SYKES and K. R. VAN HORN : *Trans. Amer. Inst. Min. Met. Eng.*, 1933, vol. 105, pp. 198–212.
16. H. ARNFELT : *Carnegie Scholarship Memoirs, Iron Steel Inst.*, 1928, vol. 17, pp. 1–20.
17. H. ARNFELT and A. WESTGREN : *Jernkontorets Ann.*, 1935, pp. 185–196.
18. E. N. KISLYAKOVA : *Zhurnal Fizicheskoy Khimii*, 1943, vol. 17, No. 2, pp. 108–114.
19. D. A. OLIVER and H. J. GOLDSCHMIDT : British Electrical and Allied Industries Research Report, N/T 41, 1946.
20. W. A. WOOD and C. WAINWRIGHT : *Phil. Mag.*, 1932, vol. 14, pp. 191–198.

ANDREW CARNEGIE RESEARCH REPORT

Diffusion of Hydrogen in Iron and Iron Alloys at Elevated Temperatures

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SYNOPSIS

The effects of chromium, nickel, and molybdenum on the rate of hydrogen permeation in iron have been investigated. Chromium has little effect on permeation in the γ range but greatly reduces the rate of permeation in the α range. Nickel and molybdenum have little effect.

The permeation equation has been re-examined ; experimental evidence leads to a general equation :

$$P = k_1 p_1^x (1 - S) \exp\left(-\frac{E}{RT}\right)$$

where P is the rate of permeation per unit area, p_1 the ingoing pressure, and T the absolute temperature ; x and S are functions of temperature such that $\frac{1}{2} < x < 1$, and $0 < S < 1$. The equation given by Richardson

$$P = K p_1^{\frac{1}{2}} \exp\left(-\frac{E}{RT}\right)$$

may be considered as a limiting case of the more general equation in which $x = \frac{1}{2}$ and $S = 0$. The variations of x and S from these values in certain circumstances are attributed to slow phase-boundary processes.

When $x \simeq \frac{1}{2}$ and $S \simeq 0$ the phase-boundary processes are believed to be fast compared with diffusion through the metal, and a time-lag method for determining D, the diffusion coefficient, has therefore been used. The values of solubility calculated from the data obtained are of the same order of magnitude as those obtained by different methods.

Introduction

THE phenomenon of gas permeation through metals was noted nearly a century ago and in recent years particular attention has been paid to the detrimental effects of hydrogen in steel—notably embrittlement and hair-line crack formation—when hydrogen is retained after solidification of the metal or introduced by prolonged soaking in hydrogen atmospheres.[1–4] A survey of the extensive literature shows that little is known concerning the fundamental factors involved in these problems, particularly the importance of the solubility and diffusivity of hydrogen in the mechanism of embrittlement and crack formation.

In studying diffusion the general equation

$$\frac{dm}{dt} = -D\frac{dc}{dx} \quad \text{.....(1)}$$

may be applied, where dm is the mass transferred across unit area in time dt at a point where the concentration gradient is dc/dx, and D is the diffusion coefficient.

Assuming that D is independent of concentration and that a steady state of flow exists, this equation gives :

$$P = D\frac{c_1 - c_2}{l} \quad \text{.....(2)}$$

where P is the rate of permeation through unit area of a slab of thickness l, and c_1, c_2 are the concentrations at $x = 0$ and $x = l$ respectively. When the diffusion of gases through metals is concerned P is readily determined, but c_1 and c_2 cannot be measured directly. It is therefore not possible to determine D from measurements of P, and practically all published work on gas diffusion through metals is concerned only with measurements of the rate of permeation. Richardson[5] (*loc. cit.*, p. 94) found from a study of the permeation of hydrogen through platinum that when the pressure on the outgoing side of the membrane was approximately zero, the process followed the equation

$$P = K\frac{1}{l}p_1^{\frac{1}{2}}\exp\left(-\frac{E}{RT}\right) \quad \text{.....(3)}$$

where K is a constant, p_1 the ingoing pressure, l the membrane thickness, T the absolute temperature, R the gas constant, and E the activation energy of permeation. When the pressure on the outgoing side is p_2 instead of zero a modified form of the equation has been applied :

$$P = K\frac{1}{l}\left(p_1^{\frac{1}{2}} - p_2^{\frac{1}{2}}\right)\exp\left(-\frac{E}{RT}\right) \quad \text{.....(3a)}$$

For permeation through membranes of given thickness at a constant temperature the equation becomes :

$$P = K'(p_1^{\frac{1}{2}} - p_2^{\frac{1}{2}}) \quad \text{.....(3b)}$$

$$\text{or if } p_2 = 0, \text{ then } P = K'' p_1^{\frac{1}{2}} \quad \text{.....(3c)}$$

Similarly for membranes of given thickness the permeation rate at fixed ingoing and outgoing pressures is given by :

$$P = K''' \exp\left(-\frac{E}{RT}\right) \quad \text{.....(3d)}$$

Two deviations from Richardson's equation have not been satisfactorily explained, *viz.* :

(1) Under isothermal conditions $P = K'' p_1^{\frac{1}{2}}$ at high but not at low pressures and the departure from the law is greater the lower the temperature.

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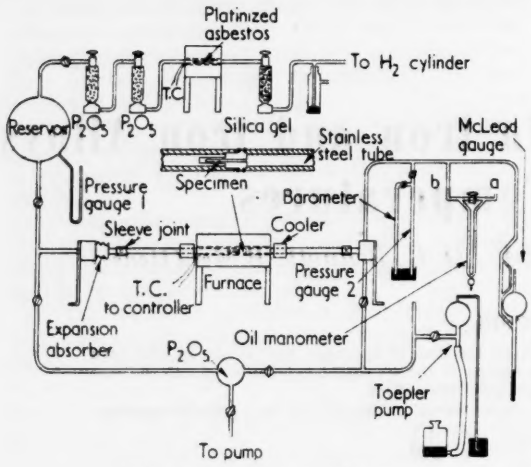

Fig. 1—Diffusion apparatus

Smithells and Ransley[6] suggested that the deviation was due to incomplete adsorption on the metal surface at low pressures. They introduced an adsorption term $(\alpha p_1)/(1 + \alpha p_1)$ in the permeation equation, giving :

$$P = K'' p_1^{\frac{1}{2}} \left(\frac{\alpha p_1}{1 + \alpha p_1} \right) \quad \ldots\ldots\ldots\ldots\ldots\ldots (4)$$

By choosing suitable values of the adsorption coefficient α, the modified equation appeared to be in good agreement with the experimental data. Fowler[7] has stated that for diatomic gases which dissociate on adsorption the term $(\alpha p_1)/(1 + \alpha p_1)$ should have been $(\alpha p_1^{\frac{1}{2}})/(1 + \alpha p_1^{\frac{1}{2}})$. Barrer[8] pointed out that adsorption is an exothermic reaction and that if incomplete adsorption is the cause of the deviation from Richardson's equation, then the deviation should be greater at higher temperatures—the reverse of observed facts. It seems, therefore, that an adsorption term in the equation is not justifiable theoretically.

(2) Observations on the permeation of hydrogen through iron under isobaric conditions by Borelius and Lindblom,[9] Ham,[10] and Ham and Rast[11] show a deviation from Richardson's equation at temperatures below about 400° C. Ham and Rast postulated an electronic change-point of iron at this temperature —a view which it is difficult to accept in the absence of any supporting evidence.

The first object of the present work was to investigate the effect of alloying on the permeation of hydrogen through iron ; the second was to investigate more fully the mechanism of gas permeation through metals in the light of the above considerations.

APPARATUS

Figure 1 shows the diffusion apparatus employed, which consists of a gas purifying system, the diffusion assembly and heating furnace, and gas collecting and measuring systems.

The specimens were tubular, having diameters of 0·330 and 0·250 in. and an effective length of $1\frac{3}{4}$ in., as shown in Fig. 2. The rate of permeation through a hollow cylinder of length h and radii r_1, r_2 is given by :

$$P = 2\pi h D \frac{c_2 - c_1}{lnr_1 - lnr_2} \quad \ldots\ldots\ldots\ldots\ldots\ldots (5)$$

where c_1, c_2 are the concentrations at $r = r_1$ and $r = r_2$ respectively.

The equivalent diffusion area is :

$$A = 2\pi h \frac{r_2 - r_1}{lnr_2 - lnr_1} \quad \ldots\ldots\ldots\ldots\ldots\ldots (6)$$

if the end effect is neglected. For the dimensions given, A is approximately 10 sq. cm.

The specimens were accurately machined in a lathe, the inner surface being finished by reaming and the outer surface by polishing with grade 0 emery paper. The variation of external diameter was usually less than 0·0005 in. and the internal diameter was almost identical with the diameter of the reamer. Specimens were welded to austenitic stainless-steel tube of 0·888 and 0·555 in. dia., the procedure being to sheath the tube over the specimen and make two separate oxy-acetylene welds. During welding, water-cooling minimized oxidation of the specimen surface.

A Pt/Pt–Rh thermocouple wired to the outside of the stainless-steel tube controlled the specimen temperature during the experiment. Although the controlling thermocouple was outside the stainless tube, the indicated temperature was never more than 2° C. different from the true specimen temperature. Consequently no separate thermocouple was used to measure the true specimen temperature. Uniformity of temperature over the length of the specimen was tested, and the variation was less than 1° C. over the temperature range employed. Permeation rates were determined by measuring the rate of increase of pressure by a McLeod gauge or an oil manometer (depending on the magnitude of the increase). As the volume of the measuring system was previously determined and the specimen dimensions were known, the rate of pressure increase could be converted into rate of permeation in c.c. (N.T.P.)/unit-area/unit-time. The pressure on the ingoing side of the specimen could be reduced to any desired value below atmospheric pressure by isolating the reservoir from the hydrogen supply and partially pumping out the hydrogen. On the outgoing side the pressure was reduced as far as possible before isolating from the pump, and during measurement the pressure never rose to more than 1 mm. Hg, even at the highest permeation rates encountered.

The effects of the back pressures were studied by admitting a certain pressure of hydrogen to the measuring side of the apparatus (indicated by pressure gauge 2, Fig. 1), and on isolating the measuring side the rate of permeation was determined by noting the pressure increase on the oil manometer. During

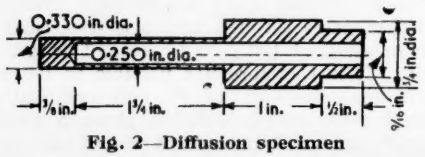

Fig. 2—Diffusion specimen

Table I

ANALYSES OF THE MATERIALS STUDIED

Material		C,%	Si,%	Mn,%	S,%	P,%	Cr,%	Ni,%	Mo,%
A	Iron	0·05	Trace	0·07	0·039	0·007	...	...	...
2	Fe–Cr	0·04	0·33	0·49	0·027	0·014	1·38	Trace	...
3	Fe–Cr	0·04	0·37	0·57	0·026	0·019	2·51	Trace	...
5	Fe–Cr	0·05	0·25	0·53	0·034	0·019	4·25	Trace	...
11	Fe–Ni	0·06	0·37	0·68	0·038	0·01	0·02	0·94	...
12	Fe–Ni	0·04	0·32	0·65	0·032	0·01	0·01	1·87	...
15	Fe–Ni	0·03	0·27	0·42	0·027	0·01	0·10	4·36	...
21	Fe–Mo	0·05	0·18	0·23	0·022	0·006	Trace	0·19	0·98
22	Fe–Mo	0·05	0·17	0·23	0·027	0·009	Trace	0·20	2·36
27	Fe–Mo	0·05	0·23	0·22	0·034	0·015	Trace	0·21	10·36

these experiments the pressure of hydrogen in the bulb (*a*) varied from 50 to 500 cm. A correction term was necessary to allow for compression, and it can be shown that if A and B are the volumes to the right and left of the manometer tap respectively, ρ_0 and ρ_m the densities of the manometer oil and of mercury, Δm the observed rise in manometer reading (in cm.) after time t, C the cross-sectional area of the oil column, p_2 the pressure in bulb (*a*) before isolating, and θ the room temperature, then Δv (the amount of gas permeating in time t (in c.c. at N.T.P.)) is given by

$$\Delta v = \left(\frac{273}{273+\theta}\right)\left(\frac{p_2}{760}\right)B\left\{\frac{2\rho_0}{p_2\rho_m}\,10\,(\Delta m) + \left[\frac{A}{B}\left(\frac{B+(\Delta m)C}{A-(\Delta m)C}\right)-1\right]\right\} \quad \ldots(7)$$

When p_2 is very small the expression

$$\left[\frac{A}{B}\left(\frac{B+(\Delta m)C}{A-(\Delta m)C}\right)-1\right]$$

may be neglected in comparison with

$$\frac{2\rho_0}{p_2\rho_m}\cdot 10(\Delta m)$$

and

$$\Delta v = \left(\frac{273}{273+\theta}\right)B\frac{20\rho_0}{p_2\rho_m}\cdot\Delta m$$

but as p_2 increases the correction term becomes more important.

EXPERIMENTS AND RESULTS

PERMEATION UNDER CONSTANT PRESSURE

($p_1 \simeq 760$ mm.Hg, $p_2 \simeq 0$)

The materials studied were an ingot iron and a series of Fe–Cr, Fe–Ni, and Fe–Mo alloys with up to 10% alloying element (*see* Table I).

The alloys were melted in a 16-lb. capacity high-frequency furnace, cast in 2-in. sq. ingots, and forged to $\frac{7}{8}$-in. dia. bars. The bars were annealed at 1000° C. for 1 hr. before the specimens were machined. After welding and assembling in the apparatus the welded joints were tested. When the welding had been satisfactorily performed a pressure lower than 0·0002 mm. Hg was obtained and there was little increase in pressure on isolating from the pump. The specimen was then heated to 1000° C. and degassed for 1 hr., after which it was soaked in pure hydrogen for 2 hr. After this the measuring

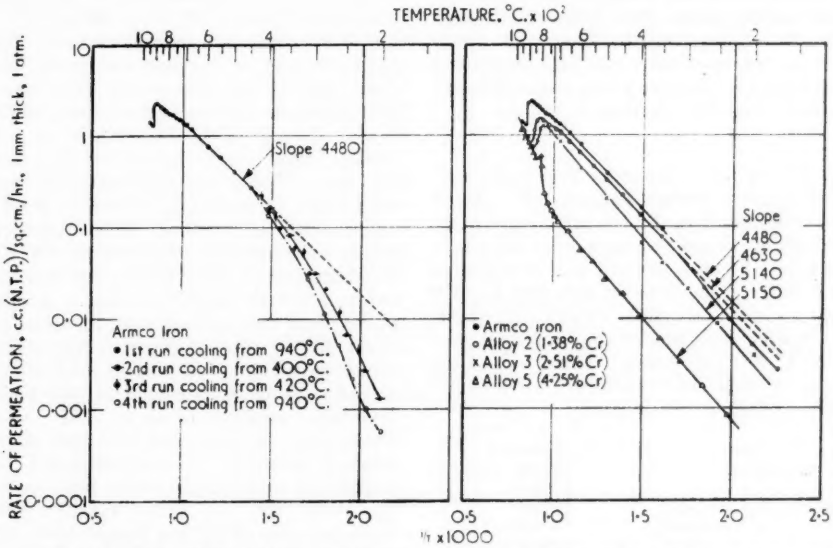

Fig. 3—Permeation isobars of Armco iron

Fig. 4—Permeation isobars of Fe–Cr alloys

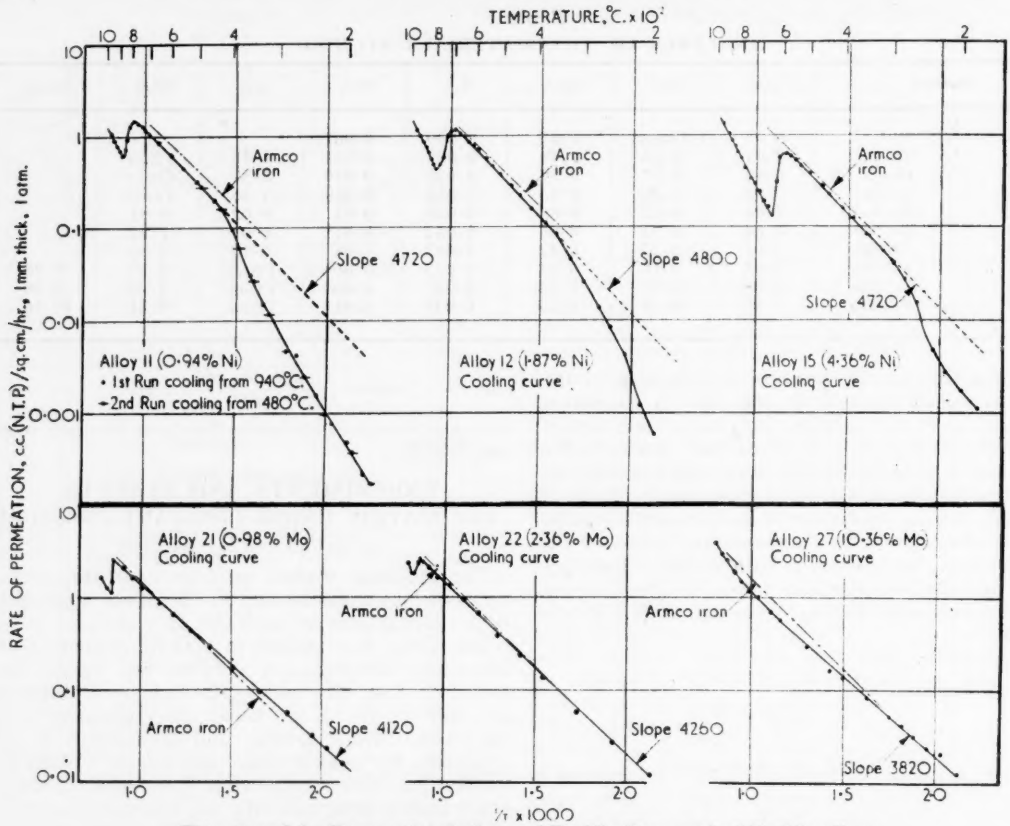

Fig. 5

Fig. 6

Figs. 5 and 6—Permeation isobars of Fe–Ni alloys and of Fe–Mo alloys

system was pumped out while the ingoing side of the membrane was under 1 atm. pressure of hydrogen.

Permeation rates at various temperatures, both on heating and on cooling, were then measured as previously described. From time to time, gas which was evolved from the specimens into the measuring system was extracted by Toepler pump and analysed ; it was always more than 98% hydrogen.

Ingot Iron

The permeation isobar of the ingot iron is given in Fig. 3 in which log P is plotted against $1/T$. Apart from the α–γ transformation and the low temperature deviations (which are discussed more fully later) the experimental points lie on a straight line as expected from equation (3*d*). The slope of this line is 4480 corresponding to an activation energy of 17,800 cal./g. mol. Another specimen of the same iron gave an E value of 17,900 cal./g. mol. These values are in fair agreement with those obtained by previous workers for the hydrogen–iron system :

Investigator	E, cal./g. mol.
Present authors	17,800
	17,900
Smithells and Ransley	19,200
Borelius and Lindblom	18,800
Ryder	22,000
Ham and Rast	17,300 ; 16,300 ; 17,700
Bennek and Klotzbach[12]	18,900

Direct plotting of permeation rate against temperature shows that the change in permeation rate at the α–γ transformation is accompanied by a hysteresis of about 18° C. Below the transformation temperature the curves coincide and there is a slight inflection at the magnetic change-point. Below about 400° C. the permeation rate becomes smaller than would be expected from equation (3*d*), as Fig. 3 shows. Repeated measurements on cooling from various temperatures below the Ac_3 point gave the full line. When the specimen was soaked for a second time in hydrogen at 940° C. for 1 hr. and the experiments on cooling were repeated, identical results were obtained above about 430° C., but below that temperature they fell on the broken line, appreciably below the first (full) line. Another specimen of the same iron gave almost identical results above about 400° C. but at lower temperatures the curve was between the full and broken curves. After a second soaking in hydrogen at 940° C. for 1 hr., the deviation occurred at about 350° C.—that is, the temperature was lowered and not raised as in the previous specimen. It was concluded that no readily established relationship exists between time of soaking above the α–γ transformation point and the characteristics of the low temperature deviation.

A second interesting feature of permeation below about 400° C. was that the results on heating and

cooling were not identical and there appeared to be a hysteresis loop in the curve. More careful study showed that this was due to the fact that the time required to establish a steady state of flow was much greater when approaching a given temperature on heating than when cooling from a higher temperature. In one experiment at 200° C., 1 hr. sufficed to set up a steady state if the specimen was cooled to this temperature, but on heating to the same temperature steady conditions were not reached even after 70 hr. Since the time required to obtain a steady state of flow on heating is so much greater than that required to measure the permeation rate, misleading results may be reported unless great care is taken. In view of the time required to obtain an equilibrium curve for permeation on heating, all the results in Fig. 3 were obtained on cooling and refer to the steady state of flow.

Fe–Cr Alloys

The results on three Fe–Cr alloys are given in Fig. 4 (which includes the ingot iron for comparison). The curves show the following features :

(*a*) In the γ range the rate of permeation is not appreciably affected by the presence of Cr, at least up to 4·25%

(*b*) In the α range the permeation rate is greatly reduced with increasing Cr and at 600° C. it is ten times smaller in the 4·25% alloy than in ingot iron

(*c*) The low temperature deviation disappears in the high Cr alloys. In the 1·38% alloy the deviation exists but is reduced in magnitude

(*d*) The apparent activation energy for permeation in the α range is increased slightly by the addition of Cr, as shown below :

Material	*E*, cal./g. mol.
Ingot iron	17,800
Alloy 2 (1·38% Cr)	18,500
Alloy 3 (2·51% Cr)	20,500
Alloy 5 (4·25% Cr)	20,600

The activation energy for permeation in the γ range, as measured from the slope of the curve obtained on the 4·25% alloy, is 37,400 cal./g. mol.

The work of Armbruster,[13] on an 18% Cr–0·3% C alloy, showed that the rate of solution of hydrogen was greatly reduced by the presence of a "barely visible film of oxide." The present results on Fe–Cr alloys may have been affected by such stable oxide films, and these may be the cause of the low permeation rates in the α range. On the other hand, a similar mechanism would be expected in the γ range, but no reduction in the permeation rate was observed.

Fe–Ni Alloys

The results are shown in Fig. 5, and the activation energies for permeation are given below :

Material	*E*, cal./g. mol. α Range	*E*, cal./g. mol. γ Range
Ingot iron	17,800	...
Alloy 11 (0·95% Ni)	18,900	36,400
Alloy 12 (1·87% Ni)	19,200	36,400
Alloy 15 (4·36% Ni)	18,900	36,000

In both the α and the γ ranges the permeation rate is not appreciably affected by the addition of Ni. The low temperature deviation is as prominent in the alloys as in ingot iron.

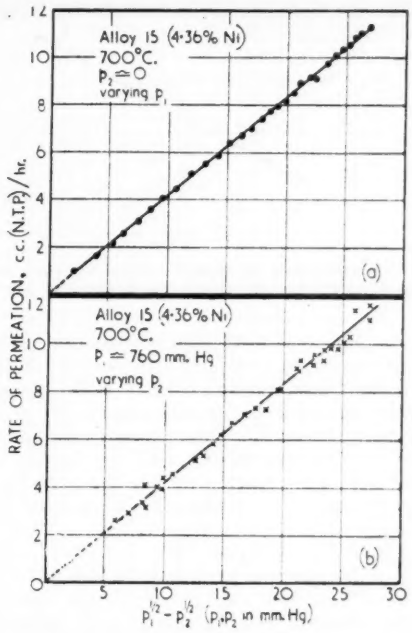

Fig. 7—Permeation at 700° C. with varying ingoing and outgoing pressures for 4·36% Ni alloy

Fe–Mo Alloys

The results are shown in Fig. 6, and the activation energies for permeation are given below :

Material	*E*, cal./g. mol.
Ingot iron	17,800
Alloy 21 (0·98% Mo)	16,500
Alloy 22 (2·36% Mo)	17,000
Alloy 27 (10·36% Mo)	15,300

Molybdenum has no appreciable effect on the permeation rate, but, as with the higher Cr alloys, no low temperature deviations have been observed.

Although the permeation data may be directly useful for problems in which the boundary conditions are similar, the changes of permeation on alloying must not be taken as an indication of changes in the diffusion coefficient D. This is clear from equation (2) since it is not known how the alloying additions affect the concentrations c_1, c_2. Previous work on solubilities indicates a small effect, but a more direct method of determining D is discussed later (p. 212).

PERMEATION AT CONSTANT TEMPERATURE

Two kinds of isothermal permeation experiment have been made : (1) With $p_2 \simeq 0$ and varying p_1, and (2) with $p_1 \simeq 760$ Hg and varying p_2. The results of both are given in Fig. 7, where the rate of permeation at 700° C. is plotted against $(p_1^{\frac{1}{2}} - p_2^{\frac{1}{2}})$ for the alloy containing 4·36% of Ni ; (*a*) was obtained by varying p_1 when $p_2 \simeq 0$ and (*b*) by varying p_2 when $p_1 \simeq 760$ mm. Hg. Since the two graphs show straight-line relationships of identical slope, the results may be represented by

$$P = K'(p_1^{\frac{1}{2}} - p_2^{\frac{1}{2}})$$

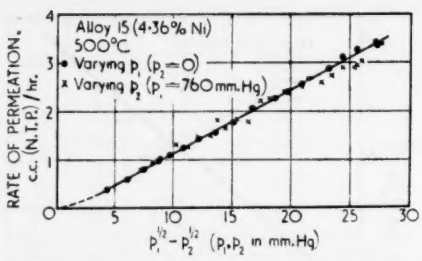

Fig. 8—Permeation at 500° C. with varying ingoing and outgoing pressures for 4·36% Ni alloy

which is the isothermal form of equation (3*a*). The straight lines pass through the origin, indicating that under these conditions there is no measurable departure from the above relationship at low pressures.

Figure 8 shows the isotherms for the same alloy at 500° C.; the results obtained by varying p_1 and by varying p_2 are plotted on the same graph. The results do not lie on a straight line passing through the origin although at higher values of $(p_1^{\frac{1}{2}} - p_2^{\frac{1}{2}})$ the relationship is linear. For the alloy containing 0·94% of Ni the results obtained when $p_2 \simeq 0$ were similar, and the low pressure deviations were greater at lower temperatures. A plot of log P against log p_1 gave an excellent straight-line relationship but x (the slope of the straight line) varied with the temperature of experiment in the manner shown in Fig. 9. At high temperatures equation (3*c*) is obeyed and the slope is $\frac{1}{2}$, but at lower temperatures the slope is much greater. These experimental results are best represented by the relation:

$$P = K_1 p_1^{x} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (8)$$

where both K_1 and x depend upon temperature. Figures 10 and 11*a* show a plot of P against p_1^x, which gives a straight line passing through the origin.

The variation of x with temperature is given in Fig. 9, in which the permeation isobar is also included. At high temperatures $x \simeq \frac{1}{2}$ and at low temperatures $x \simeq 1$, and the low temperature deviation of the isobar occurs at the temperature at which the departure of x from $\frac{1}{2}$ becomes appreciable.

The isothermal results may be used to test the modification of Richardson's equation suggested by Smithells and Ransley (equation 4). If their assumptions are correct a straight line should result when $(p_1^{\frac{3}{2}}/P)$ is plotted against p_1 (using the adsorption term $(\alpha p_1)/(1 + \alpha p_1)$) or when p_1/P is plotted against $p_1^{\frac{1}{2}}$ (using the adsorption term $(\alpha p_1^{\frac{1}{2}})/(1 + \alpha p_1^{\frac{1}{2}})$. Figures 11*b* and 12 show typical results of such plots; at low temperatures the agreement is poor.

The influence on permeation rate of variations in outgoing pressure p_2 was also investigated in the temperature range where x is appreciably greater than $\frac{1}{2}$. The results given in Fig. 13 refer to the 0·94% Ni alloy at 320° C. When the results are interpreted in terms of a 'back-permeation' rate P_2 (defined by $P = P_1 - P_2$, where P is the observed permeation rate and P_1 is the permeation rate when $p_2 \simeq 0$), then the relationship between P_2 and the outgoing pressure p_2 is

$$P_2 = K_2 p_2^{y} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (9)$$

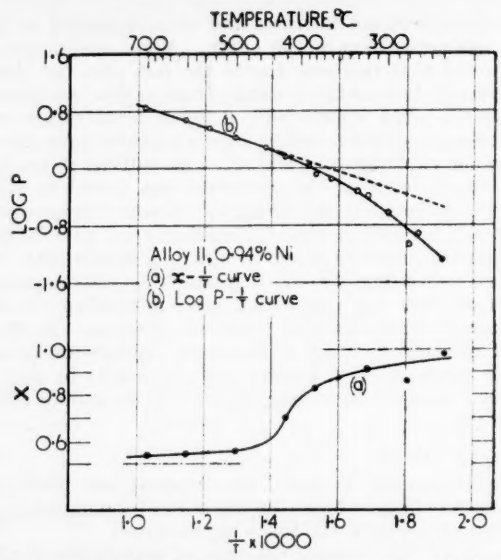

Fig. 9—(*a*) Variation of x with temperature. (*b*) Permeation isobar

where K_2 and y are functions of temperature and for the results given in Fig. 13, $y = 0{\cdot}555$. These results, when combined with the results on the same alloy given in Fig. 11*a*, suggest the equation:

$$P = K_1 p_1^{x} - K_2 p_2^{y} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (10)$$

DISCUSSION

A consideration of the isothermal and isobaric permeation results given above gives an empirical equation:

$$P = (K_1 p_1^{x} - K_2 p_2^{y})(1 - S) \exp\left(-\frac{E}{RT}\right) \ldots\ldots 11$$

where K_1 and K_2 are constants and x, y, and S are functions of temperature. Richardson's equation may be regarded as a limiting case of the above in which $K_1 = K_2 = K$, $x = y = \frac{1}{2}$, and $S = 0$.

The derivation of Richardson's equation involves the assumptions that: (*a*) the gas molecules dissociate on entering the metal, and (*b*) the gas concentrations at the surfaces of the permeation membrane are equilibrium concentrations.

The first assumption seems to be well supported

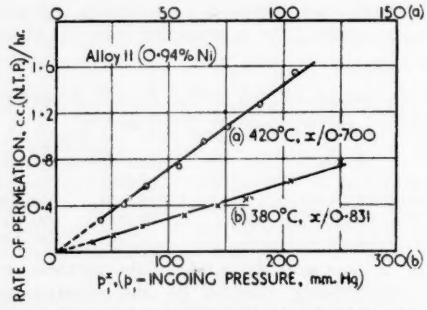

Fig. 10—Permeation isotherms for 0·94% Ni alloy, with permeation rate plotted against p_1^x

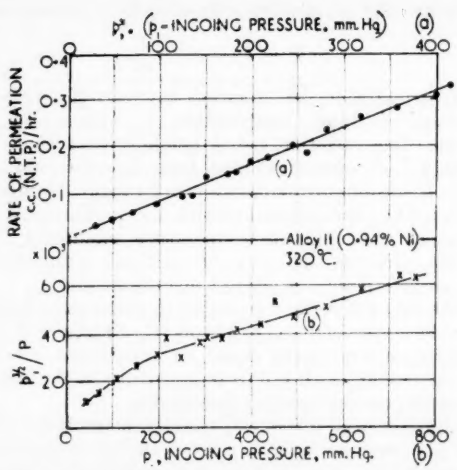

Fig. 11—Permeation isotherms for 0·94% Ni alloy at 320° C. (*a*) *P* plotted against p_1^x; (*b*) the same results plotted to test Smithells and Ransley's assumptions

by the available evidence, but the second is valid only if the phase-boundary processes (*e.g.*, adsorption and solution) are fast, compared with permeation through the metal. If sufficiently rapid, these processes will be capable of supplying more material to the surface layers than is removed by the diffusion processes, and the concentration of dissolved gas at the surface will be the equilibrium concentration at that pressure. On the other hand, if the diffusion process is capable of removing more material than the surface processes supply, then the concentration at the surface will fall below the equilibrium value. Similarly, at the outgoing surface the concentration of dissolved gas will be the equilibrium concentration if the surface processes are more rapid than the diffusion process, but it will be higher than the equilibrium value if the surface processes are slow.

Since there is no direct experimental support for the assumption that the surface concentrations are equilibrium values, an examination of the possible effects of slow surface processes may lead to an

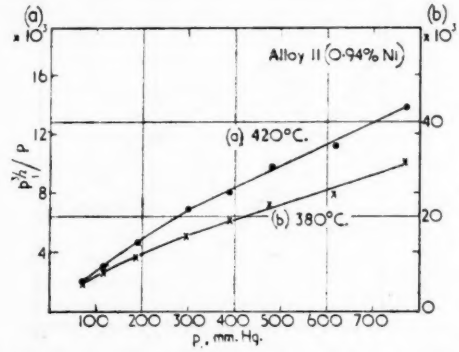

Fig. 12—Permeation isotherms for 0·94% Ni alloy, plotted to test Smithells and Ransley's assumptions

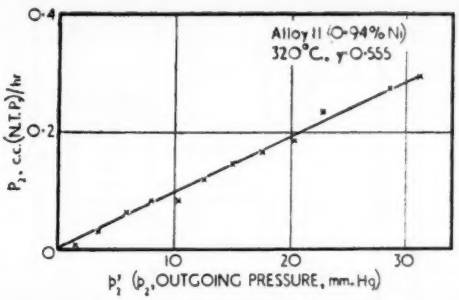

Fig. 13—Variation of 'back permeation' rate P_2, with outgoing pressure, p_2

explanation of the observed departures from Richardson's equation.

The problem has previously been investigated by Wang,[14] Smithells and Ransley,[15] and Barrer[16] (*loc. cit.*, p. 353) from the point of view of studying whether the permeation rate will be finite at very high pressures. Barrer[16] (*loc. cit.*, p. 148) considered that the surface processes could be adequately described by six equations involving six velocity constants and including the processes of adsorption, solution from the adsorbed layer, and a process in which one atom was dissolved and one was adsorbed when a molecule struck the surface. His treatment is admirably complete, but it is not easy to simplify his expressions to give an explicit relation between the permeation rate P and the gas pressure p_1. The dependence of concentration gradient, and hence of permeation rate upon pressure, is very complex when the transfer of matter by surface processes takes place at a rate comparable with that of the diffusion process.

However, if we simplify the model of the process and consider only direct solution from the gaseous phase we may examine the effects of the speed of this process on the permeation rate. If we consider a membrane bounded by the surfaces $x = 0$ and $x = l$ at which the gas pressures are p_1 and p_2 respectively, and the concentrations of dissolved gas are c_1 and c_2 respectively, and if c_s denotes the saturation concentration of dissolved gas (*i.e.*, the limiting concentration approached as p_1 and $p_2 \to \infty$), then we may write for $x = 0$:

$$\left.\begin{aligned} &\text{Rate of solution} && \frac{dm_1}{dt} = k_1 p_1 \left(1 - \frac{c_1}{c_s}\right)^2 \\ &\text{Rate of evaporation} && \frac{dm_2}{dt} = k_2 c_1^2 \end{aligned}\right\} \quad \ldots(12)$$

and for $x = l$:

$$\left.\begin{aligned} &\text{Rate of evaporation} && \frac{dm_3}{dt} = k_2 c_2^2 \\ &\text{Rate of solution} && \frac{dm_4}{dt} = k_1 p_2 \left(1 - \frac{c_2}{c_s}\right)^2 \end{aligned}\right\} \quad \ldots(13)$$

and inside the metal :

$$\text{Rate of permeation} \quad P = D\frac{c_1 - c_2}{l} \qquad \ldots\ldots\ldots(14)$$

In the steady state of flow :

$$P = \frac{dm_1}{dt} - \frac{dm_2}{dt} = \frac{dm_3}{dt} - \frac{dm_4}{dt}$$

The available evidence suggests that under pressures of 1 atm. the concentration attained is so small

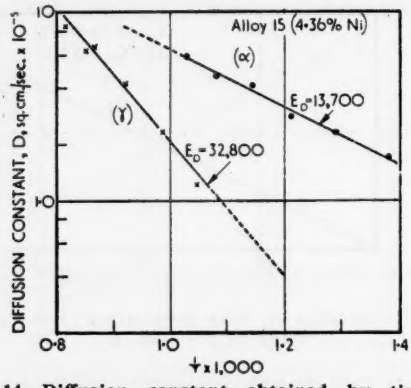

Fig. 14—Diffusion constant obtained by time-lag method for 4·36% Ni alloy

compared with c_s that the term $(1 - c_1/c_s)$ may be taken as approximately equal to 1. If, further, we consider only the cases where $p_2 \simeq 0$ and we ignore the term

$$k_1 p_2 \left(1 - \frac{c_2}{c_s}\right)^2$$

then we may eliminate c_1 and c_2 from these equations and obtain:

$$P\left\{1 + \left(\frac{l\sqrt{P}\sqrt{k_2}}{D} + 1\right)^2\right\} = k_1 p_1 \quad \text{.....(15)}$$

The relation between P and p_1 then depends on the magnitude of the term $(l\sqrt{P}\sqrt{k_2})/D$ for if $(l\sqrt{P}\sqrt{k_2})/D \gg 1$, then $l^2P^2k_2/D^2 \simeq k_1p_1$ or:

$$P \simeq \frac{D}{l}\sqrt{\frac{k_1}{k_2}} \cdot p_1^{\frac{1}{2}} \quad \text{.....(16)}$$

but if $(l\sqrt{P}\sqrt{k_2})/D \ll 1$, then:

$$P \simeq \frac{k_1}{2} p_1 \quad \text{.....(17)}$$

Now, in the terms $(l\sqrt{P}\sqrt{k_2})/D$ or $lP/D.\sqrt{(k_2/P)}$, $lP/D = c_1 - c_2$ (*see* equation 2), that is, the difference of concentration across the membrane, and $\sqrt{(k_2/P)}$ expresses the relative magnitudes of the surface rate of evaporation and the rate of permeation through the membrane. If at high temperatures the surface reactions are fast, k_2 will be large compared with P, and the concentrations at the two surfaces will be the equilibrium concentrations, *i.e.*, $c_1 - c_2$ will not be very small for moderate values of p_1 and therefore equation (16) will be valid. If, however, at low temperatures the surface processes are slow, then in addition to k_2 being small compared with P, the value of c_1 and c_2 will differ from the equilibrium values as indicated above and the term lP/D may become small, and equation (17) may apply.

For the intermediate cases where surface processes and diffusion processes take place at similar rates, it is not easy to determine the relationship between P and p_1, although experiment shows that it is of the form $P \propto p_1^x$ where $\frac{1}{2} < x < 1$.

If this approximate relation is accepted, the variation of x with temperature will indicate the change of the relative magnitudes of the surface and diffusion processes. In the absence of slow surface processes the permeation rate may be expressed as:

$$P = P_0 \exp\left(-\frac{E}{RT}\right)$$

If, in addition, the relation between the velocity constant k_2 and temperature is exponential in nature, the variation being much greater than the variation of permeation rate, then the condition that $k_2 \sim P$ will hold over only a small range of temperature, as the experimental results show. Furthermore, since at the highest temperature examined x is slightly greater than $\frac{1}{2}$, even at these temperatures the surface processes are not instantaneous.

The effect of surface processes in permeation isobars may be expressed by a factor $(1-S)$ where S is a 'slowing-down' term equal to zero when $x = \frac{1}{2}$, but increasing with fall in temperature as the surface processes assume greater importance.

Alloying may affect the relative magnitudes of k_2 and D in such a way as to remove the low temperature discontinuity in the isobars or to reduce the temperature of its occurrence below the range of this investigation. Surface condition may also affect k_2 so that the discontinuity may occur at a different temperature after heating above the transformation point, as this may produce a slight change in surface condition.

MEASUREMENT OF THE DIFFUSION COEFFICIENT

Permeation data alone are of limited value; of more fundamental interest in various problems is the diffusion coefficient D. Daynes[17] and Barrer[18] worked out a method for determining D in which a simple relation holds between D and the time-lag required to set up a steady state of flow when the diffusing gas is suddenly admitted to one side of a previously degassed membrane. The method is applicable only in the absence of slow surface reactions when the diffusion within the membrane is the factor controlling the flow. It has been used for the diffusion of gases through rubbers and high polymers and for the diffusion of nascent hydrogen through iron at near-room temperature. From considerations given in the preceeding section, the authors have concluded

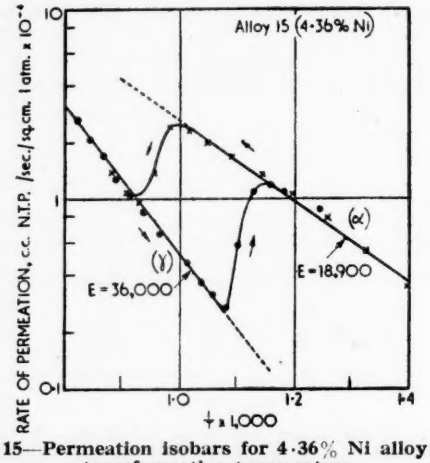

Fig. 15—Permeation isobars for 4·36% Ni alloy near transformation temperature

that the surface processes are fast when x (in equation 8) is approx. $\frac{1}{2}$. The time-lag method for determining D may therefore be used in the temperature range for which this condition holds. If c_0 is the initial concentration of gas in the permeation membrane, *i.e.*, $c = c_0$ for $0 \leqslant x \leqslant l$ and $t = 0$, then the sudden introduction of gas to one side of the membrane while the other is kept as near as possible to zero pressure, will result in the gradual building up of a concentration gradient within the metal until equilibrium conditions are reached. If the amount of gas liberated from the outgoing surface of the specimen is measured at various times and a curve is plotted, L, the intercept made by the linear portion of the curve on the time axis, may be determined. The relation between L and D given by Barrer (*loc. cit.*) is

$$L = \frac{l^2}{D} \cdot \frac{1}{(c_1 - c_2)}\left[\frac{c_1}{6} + \frac{c_2}{3} - \frac{c_0}{2}\right] \quad \text{.....(18)}$$

When the specimen is degassed at the experimental temperature and the surface processes are rapid, $c_0 = c_2$ and the relation reduces to

$$L = \frac{l^2}{6D} \quad \text{.....(19)}$$

The results obtained using this relation are shown in Fig. 14, in which log D is plotted against $1/T$ for the alloy containing 4·36% of Ni. The values of L were about 1 min. and were reproducible. These values were calculated from the experimental points using a regression technique. Figure 15 shows the permeation curves in the same temperature range. Although this method is not advocated as a means of determining the gas solubility, a calculation based on Figs. 14 and 15 shows that the solubilities obtained are similar to those obtained by very different methods[4] (*loc. cit.*, p. 159).

Temperature °C.	Solubility in c.c. (N.T.P.)/100 g., at 760 mm. Hg Time-Lag Method Fe–4·36% Ni Alloy	Conventional Method Fe	Ni
700	5·1	1·9	6·6
650	4·7		
600	4·3	1·3	5·5
550	3·9		
500	3·4	0·9	4·5

Equation (19) is applicable only when the permeation membrane is of plate form ; for cylindrical tubes the relation is [19] :

$$L = \frac{2}{D}\sum_{1}^{\infty} \frac{1}{\alpha_n^2} \cdot \frac{J_0(\alpha_n r_2) \cdot J_0(\alpha_n r_1)}{J_0^2(\alpha_n r_1) - J_0^2(\alpha_n r_2)} \cdot \log \frac{r_1}{r_2} \quad \text{.....(20)}$$

where J_0 is a Bessel function of the first kind and zero order, α_n is the nth positive root of $U_0(\alpha r_2) = 0$, and U_0 is defined by

$$U_0(\alpha r_r) = J_0(\alpha r)H_0^1(\alpha r_2) - J_0(\alpha r_2)H_0^1(\alpha r)$$

where H_0^1 denotes a Bessel function of the third kind and α is a root of $U_0(\alpha r_2) = 0 = U_0(\alpha r_1)$. For a tube of the dimensions of the present specimen, equation (20) would give values for the diffusion coefficient D about 10% higher than those given by equation (19). In considering gas diffusion through metals, the diffusion coefficient D has generally been assumed to be independent of concentration. This is true within the concentration range investigated, as shown by Figs. 7 and 8, where the rate of permeation is not affected by variations of p_1 or p_2 as long as $(p_1^{\frac{1}{2}} - p_2^{\frac{1}{2}})$ is constant. Further, in the time-lag method the value of D obtained is independent of the value chosen for the ingoing pressure p_1. The following results were obtained with the 4·36% Ni alloy at 700° C. :

Pressure (p), mm. Hg	Diffusion Coefficient (D), cm.²/sec. Time-Lag Method × 10^{-6}
71	5·9
150	6·1
238	6·1
313	5·9
396	6·1
482	5·7
573	5·8
667	5·9
758	5·9

Since the pressure range (and hence the concentration range) covered is rather narrow, the conclusion must not be extended to high pressures without further investigation.

Acknowledgments

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References

1. J. H. Andrew and others : *J. Iron Steel Inst.*, 1942, No. II, pp. 193, 203 ; 1946, No. I, p. 67 ; 1947, vol. 156, p. 208.
2. C. Sykes, H. H. Burton, and C. C. Gegg : *Ibid.*, 1947, vol. 156, p. 155.
3. C. A. Zapffe and C. E. Sims : *Trans. Amer. Inst. Min. Met. Eng.*, 1941, vol. 145, p. 225.
4. H. H. Uhlig : *Ibid.*, 1944, vol. 158, p. 183.
5. C. J. Smithells : " Gases and Metals." Chapman and Hall, 1938. Also *see* O. W. Richardson, J. Nicol, and T. Parnell : *Phil. Mag.*, 1904, vol. 8, p. 1.
6. C. J. Smithells and C. E. Ransley : *Proc. Roy. Soc.*, 1935, vol. 150*A*, p. 172.
7. R. H. Fowler : *Proc. Camb. Phil. Soc.*, 1935, vol. 31, p. 260.
8. R. M. Barrer : " Diffusion in and through Solids," p. 172. Cambridge University Press, 1941.
9. G. Borelius and S. Lindblom : *Annalen der Physik*, 1927, vol. 82, p. 201.
10. W. R. Ham : *Trans. Amer. Soc. Metals*, 1937, vol. 25, p. 536.
11. W. R. Ham and W. L. Rast : *Ibid.*, 1938, vol. 26, p. 886.
12. H. Bennek and G. Klotzbach : *Technische Mitteilungen Krupp—Forschungberichte*, 1941, vol. 4, p. 47.
13. M. H. Armbruster : *J. Amer. Chem. Soc.*, 1943, vol. 65, pp. 104–154.
14. J. S. Wang : *Proc. Camb. Phil. Soc.*, 1936, vol. 32, p. 657.
15. C. J. Smithells and C. E. Ransley : *Proc. Roy. Soc.*, 1936, vol. 157*A*, p. 292.
16. R. M. Barrer : *Phil. Mag.*, 1939, vol. 28.
17. H. A. Daynes : *Proc. Roy. Soc.*, 1920, vol. 97*A*, p. 286.
18. R. M. Barrer : *Trans. Faraday Soc.*, 1939, vol. 35, p. 628.
19. R. M. Barrer : *Ibid.*, 1940, vol. 36, p. 1235.

The Hot-Torsion Test for Assessing Hot-Working Properties of Steels

By D. E. R. Hughes, B.Sc., Ph.D.

SYNOPSIS

A study has been made of the hot-torsion test to assess its suitability for determining the hot-workability of steels. The design and construction of a testing machine are described, and a suitable experimental technique has been developed.

Tests on mild-steel and, later, on high-carbon chromium steel specimens, to determine the effect of testing temperature and speed, over the ranges 950–1400° C. and 12–600 r.p.m. respectively, on the number of revolutions to failure and on the torque, showed, in all cases, a well-marked maximum in the curves connecting revolutions to failure with temperature. Except at very low testing speeds (12–50 r.p.m.) for the mild-steel specimens, the temperature at the maximum is that found to be best for the rotary piercing operation in the manufacture of seamless steel tubes.

Examination of mild-steel and high-sulphur mild-steel specimens that had been deformed only part of the way to fracture showed that the outer regions underwent very heavy deformation, and that a flow effect at right angles to that of the original rod ultimately developed. The longitudinal tensile stress probably became an important factor influencing failure. Specimens cut transversely from a mild-steel billet gave very much lower values for revolutions to failure than longitudinal ones from the same billet.

As a result of this work, standardized conditions for testing carbon and low-alloy steels have been established.

Introduction

ALTHOUGH the effect of such factors as composition, heat-treatment, etc., on the properties of forged metal components is well known, the best conditions for the hot-working operation itself are still determined by rule-of-thumb. Much time and material would be saved if the costly and time-consuming works trials on new or modified steels were prefaced by suitable laboratory tests for hot-workability.

The mechanical properties of a metal or alloy, under appropriate conditions of temperature and rate of deformation, must be closely related to its hot-working characteristics. Recent extensive researches on these properties at elevated temperatures have dealt mainly with conditions under which the magnitude, and the rate of increase of deformation were restricted to very low values. Even in the so-called 'short time' tests, the speeds of deformation are almost invariably very much lower than those of industrial hot-working processes. Such tests are intended for material used at high temperatures normally below the forging range, when high hot strength is required.

There are few publications dealing with laboratory tests for hot workability. Most of them describe the modification of fairly standard forms of mechanical test. Portevin and his colleagues[1, 2] have investigated a number of laboratory tests for forgeability of steels and some non-ferrous alloys. The results of the dynamic tests (impact bending and impact compression) correlated with practical experience better than those of the static tests. The impact compression test, although used by a number of workers,[3–7] does not seem capable of indicating the best temperature for hot working a given material.

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Itihara[8] has published a series of papers on the impact torsion test, that deals mainly with steels, and includes temperatures in the forging range. His apparatus consisted essentially of a large flywheel which, after acceleration to a high speed, was suddenly coupled to the test piece. Static torsion tests gave results for the temperature of maximum deformation before fracture that differed considerably from those of the impact tests. Itihara concluded that the forging properties of steels could not be determined from a static test.

From results on many steels, Clark[9] has concluded that hot-torsion tests indicate reliably the best temperature for hot working. The number of revolutions to failure when related to temperature normally showed a maximum; the temperature at which it occurred was considered to be the optimum forging temperature. The test piece employed by Clark, a 22-in. forged rod, had a diameter of $\frac{5}{8}$ in. throughout, with no central reduced portion. As it is unlikely that deformation would be limited to the hottest (central) portion the results from such a test piece can have little significance. A very similar test, to which the same criticism applies, has been described by Ihrig.[10]

Bloom, Clark, and Jennings[11] consider that although it has limitations in the form used by Clark and Ihrig, the hot-torsion test is still valuable because it gives an approximate measure of workability at different temperatures. They employed the test to study the relation between the structure and hot-workability of stainless steel, and found that austenite and ferrite, when present together, apparently caused poorer workability than when only the predominant phase for a given alloy composition was present.

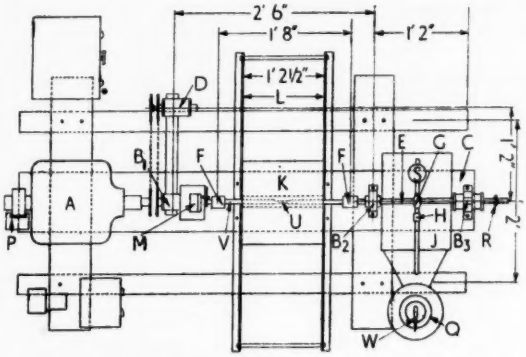
Initially, the present investigation consisted of the development of a suitable testing technique for use with the hot-torsion test, as it was thought that this would give better results than other forms of test. The

work involved the design and construction of a hot-torsion testing machine, and the determination of the best form of test piece. Torsion tests were then carried out to determine the effects of variations in testing speed, test-piece diameter, and testing temperature. Records were obtained of the torque and of the number of revolutions needed to break the test piece. All the test pieces were prepared from $\frac{3}{4}$-in. dia. rod of the same cast of mild steel. Micro-examination was carried out on a number of the broken test pieces, and on some which had been subjected to various amounts of twisting, insufficient to break them. A high-sulphur free-cutting steel was also used so that the changes in form and distribution of the inclusions could be followed. The temperature rise and the tensile force produced by the twisting were also determined.

The application of the hot-torsion test to a high-carbon chromium steel was then investigated in less detail. The test pieces were again prepared from $\frac{3}{4}$-in. dia. rod. This work was designed to ascertain how far the previous results agreed with practical experience, and whether the test conditions found to be suitable for mild steel would be suitable for different materials. Finally, pieces cut transversely and longitudinally from a mild-steel billet were tested.

APPARATUS AND TESTING PROCEDURE

A scale drawing of the apparatus is shown in Fig. 1. It is driven by an electric motor, a range of speeds from 12–600 r.p.m. being obtained by means of a countershaft and change wheels. An electrical record of the torque variations during each test is obtained by means of the slide wire on the weighing machine. When the test piece fails, the motor is automatically stopped. The electric furnace for heating the test



A. Electric Motor (1·5 h.p., with built-in reduction gear for 100 r.p.m.)
$B_{1,2,3}$. Main bearings
C. Channel section main girder
D. Countershaft bearing
E. Extension spindle
F. Quick release coupling
G. Torque arm
H. Adjustable pressure block
J. Platform of weighing machine
K. Furnace
L. Furnace slide rails
M. Revolution counter
N. Relay for automatic stop
P. Magnetic brake
Q. Slide wire on dial of weighing machine
R. Collar to prevent shortening of test piece
S. Counterweight on torque arm
U. Test piece
V. Extension to test piece
W Pointer carrying sliding contact.

Fig. 1—Torsion testing apparatus

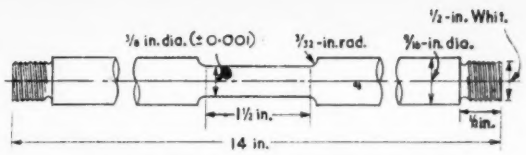


Fig. 2—Standard steel torsion test piece

piece is platinum wound, and the 12-in. tube has a $1\frac{3}{4}$-in. bore, with a liner tube of $1\frac{3}{8}$-in. bore. Approximate measurements of the tensile force set up in the restrained test piece are given later.

Plain rods, with no central reduced portion to form the actual test length, were not considered suitable as test pieces because they did not enable the deformation to be confined to a definite length that could be maintained at a uniform known temperature in the furnace. After preliminary experiments, the dimensions shown in Fig. 2 were adopted as standard for material in rod form. For tests on transverse slices of a tube-making billet, only 5-in. lengths could be used, necessitating special couplings of a Ni–Cr heat-resisting alloy, and a larger diameter shoulder ($\frac{11}{16}$ in.) for the test piece ; the dimensions of the test length were unaltered.

A temperature check of both types of test piece, made at temperatures from 950° to 1350° C., showed that the maximum variation along the test length was 4° C. at 1350° C., and less at the lower temperatures. The thermocouple used for temperature measurement during the actual test was spot-welded to the back (non-rotating) shoulder of the test piece, appropriate correction being made for the slight difference between the temperature here and at the centre of the test length.

Before a test, the thermocouple and extension pieces were attached, and the assembly preheated to about 300° C., the furnace being maintained at 800–900° C. between tests. Immediately the test piece was in position the ends of the furnace tube were plugged with asbestos wool. During heating, dry nitrogen was passed into the furnace at a rate of $2\frac{1}{2}$–3 l./hr. to minimize oxidation. The total heating time varied from about 20 min. at 950° C. up to 1 hr. at 1350° C. In each case the temperature was held constant for 5 min. before starting the test. During testing an appreciable temperature rise in the specimen sometimes occurred. Attempts were made to determine this rise more accurately using a test piece with an internal thermocouple.

MATERIALS

The test pieces for the main series of tests on mild

Table I
ANALYSES OF STEELS

Element	Steel *R*	Steel *FC*	Steel *X*
Carbon	0·10%	0·11%	1·14%
Silicon	0·22%	0·08%	0·23%
Manganese ...	0·37%	0·94%	0·48%
Sulphur ...	0·45%	0·217%	0·031%
Phosphorus ...	0·013%	0·025%	0·034%
Chromium ...	0·02%	...	1·38%
Nickel	0·08%	...	0·18%

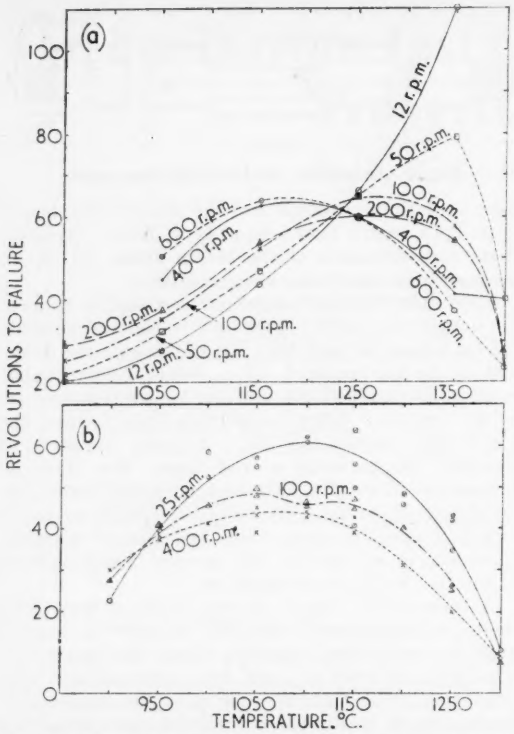


Fig. 3—Effect of testing temperature on revolutions to failure in torsion (standard $\frac{3}{8}$-in. dia. test pieces). (*a*) Steel *R* ; (*b*) steel *X*

steel were machined from $\frac{3}{4}$-in. dia. hot-rolled rod from a selected cast (steel *R*). The free-cutting steel (steel *FC*) used to study the mechanism of deformation, was supplied in the form of $\frac{5}{8}$-in. dia. bright-drawn rod. The high-carbon chromium steel (steel *X*) was taken from a cast produced for tube manufacture ; it was supplied in the form of $\frac{3}{4}$-in. dia. rod. The analyses are given in Table I.

For the tests on transverse billet slices, a 5-in. square tube-billet of mild steel, as used in the push-bench process, was employed. This steel was not analysed, but microscopical examination showed that the structure varied very little over the cross-section, and that there was no appreciable segregation or excessive amount of non-metallic matter.

EXPERIMENTAL RESULTS

Effect of Temperature and Testing Speed

The effect of temperature on the number of revolutions to failure at different testing speeds for steels *R* and *X* is shown in Figs. 3*a* and *b* respectively. Each of the points plotted is the mean of at least two, and sometimes three or four determinations. It will be seen that each of the curves, except the one for steel *R* at 12 r.p.m., passes through a maximum at a particular temperature. In the case of steel *R*, this temperature is related to testing speed, but for steel *X* the maximum occurs at about the same temperature for each of the three testing speeds used.

A complete record of the torque variations during each test was obtained, and selected examples of such records for steel *R* are shown in Fig. 4. At the lower testing speeds (12, 25, and 50 r.p.m.) the torque remained substantially constant throughout, as indicated by diagrams *a*, *b*, and *c*. With the higher speeds, however, the torque generally decreased as the test proceeded (diagrams *d*, *e*, and *f*), this effect being less marked when the temperature was raised. For comparison, the torque at 10 revs. was selected as a standard basis, because at the higher testing speeds the initial oscillations of the recorder made a reliable reading impossible at an earlier stage. The effect of testing temperature on the torque at different testing speeds for steels *R* and *X* is shown in Figs. 5*a* and *b*, respectively. The points plotted are the mean of two or more determinations ; the reproducibility was generally rather better than for revolutions to failure.

Effect of Test-Piece Diameter

The effect temperature has on the number of revolutions to failure at different speeds for test pieces of $\frac{1}{2}$-in. and $\frac{1}{4}$-in. dia. is shown in Figs. 5*c* and *d*, respectively. These curves closely resemble those obtained for the standard test pieces of $\frac{3}{8}$-in. dia. (Fig. 3*a*).

Examination of Broken Test Pieces

A number of broken test pieces in both steels were sectioned along the centre axis and examined microscopically. Low-power examination sometimes showed internal break-up or cavitation, this being very marked in steel *X*. Sections with this cavitation are shown in Figs. 7 and 8 for steels *R* and *X*, respectively. The amount of cavitation is apparently related, particularly in steel *R*, to the number of revolutions which the test piece has withstood ; at 1350° C. this number was markedly influenced by testing speed. The greater severity and the transverse nature of the internal break-up in steel *X* is shown. There is a marked tendency for the breaks to occur in two lines parallel to the longitudinal axis of the test piece ; this effect was also noted to a lesser extent with steel *R*.

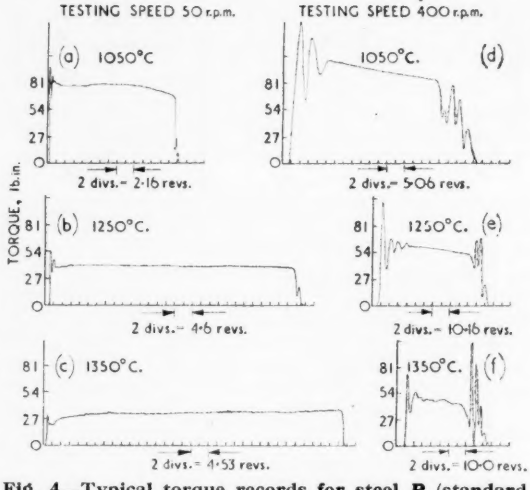


Fig. 4—Typical torque records for steel *R* (standard $\frac{3}{8}$-in. dia. test pieces)

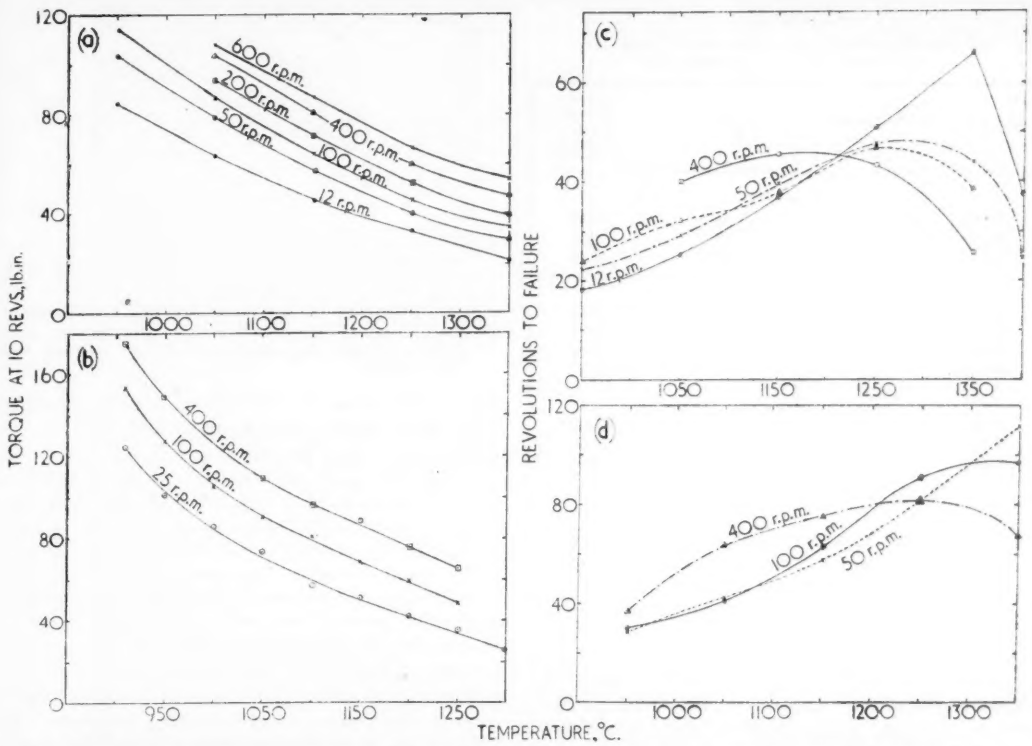


Fig. 5—Effect of testing temperature (*a*) on the torque at 10 revs. for steel *R* (standard test pieces) ; (*b*) on the torque at 10 revs. for steel *X* (standard test pieces) ; (*c*) on revolutions to failure in torsion for ½-in. dia. test pieces (steel *R*) ; (*d*) on revolutions to failure in torsion for ¼-in. dia. test pieces (steel *R*)

Examination at high magnification showed that in steel *R* the cavities were isolated holes, often elongated in the transverse direction. The cavities and the final fracture were apparently unrelated to the microstructure, and no sign of intercrystalline cracking or hot shortness appeared below 1400° C. ; typical structures are shown in Figs. 9*a* and *b*. Most of the test pieces in steel *X* had cooled fairly rapidly in air from the testing temperature, and had an almost entirely martensitic microstructure. Some contained cracks which had obviously occurred at a relatively low temperature, during the martensite transformation. At testing temperatures of 1200° C. and above, however, there were also intercrystalline cracks associated with the main fracture. A typical unetched crack, illustrated in Figs. 9*c* and *d*, showed by its oxide content that it had formed at a high temperature. At 1300° C. partial fusion had occurred.

Interrupted Tests on Steel *R*

Deformation of the test piece was stopped, after ¼, ½, and ¾ of the number of revolutions needed to cause fracture, in a few tests on steel *R*. Cavitation could be detected after about 40 revs. ; thus, under most testing conditions, the only specimens showing the effect were those which had received three-quarters of the deformation needed to cause failure. The cavities, even at the earliest stages, were nearly always elongated transversely. Except at the centre line of the section, the inclusions in the steel were broken up into extremely small particles, but there was no evidence that they were associated with the formation of cavities or with any other feature of the microstructure.

Interrupted Tests on Steel *FC*

To fracture test pieces of this steel at 100 r.p.m. the revolutions required were 13½ at 1050° C. and 21½ at 1250° C. Sulphur prints from longitudinal sections of test pieces interrupted before fracture (*see* Fig. 10) show that in all cases the centre line of the test length is light in colour, whilst the outer regions are considerably darker. This difference between the centre and outer zones becomes more distinct as the amount of deformation increases, and the lighter line in the centre becomes narrower. In the test pieces which were very close to failure (Figs. 10*e* and *i*), transverse markings, approximately equal in number to the revolutions, can be seen along the centre line. Similar markings not sufficiently pronounced for photography, were sometimes observed on steel *R*. The transverse breaks visible in Fig. 10*i* show that this test piece was on the point of fracturing, and that fracture commences from the outside.

The effects after testing at the two temperatures were found to be similar, and the following observations refer to the test pieces deformed at 1250° C. Most of the sections were examined unetched, in order to follow changes in the form and distribution

Table II

MEASUREMENT OF TENSILE FORCE ACCOMPANYING TORSION

Test-piece	Temperature, °C.	Tension, lb. wt.	Average Tensile Stress, lb./sq. in. (load/area).
	Testing Speed 25 r.p.m.		
*R*250	1350	60	545
*R*249	1250	95	864
*R*248	1050	170	1545
	Testing Speed 100 r.p.m.		
*R*244	1350	115	1046
*R*239	1250	150	1363
*R*242	1050	220	2000
*R*243	950	300	2727
	Testing Speed 400 r.p.m.		
*R*247	1350	110	1000
*R*245	1250	180	1636
*R*246	1050	290	2636

of the inclusions. It was found that as the number of revolutions increased from 2·5 to 19, the inclusions along the centre line gradually turned until they were at an angle of 45° to the longitudinal axis, as shown in Figs. 11*a* and *b*. The inclusions in the outer zones showed, as would be expected, more evidence of deformation of the steel. After 2·5 revs. (Fig. 11*c*) they were completely broken up into small globular particles, and had lost all trace of their longitudinal orientation. At later stages this break-up was even more pronounced (Fig. 11*d*), and after 19 revs. the small particles had become elongated in a transverse direction (Fig. 11*e*). The test piece which had received 19 revs. and was on the point of failure, contained a number of small transverse breaks near the outside in addition to the large breaks visible in Fig. 10*i*. Some of these appeared to be associated with sulphide inclusions, as shown in Fig. 11*f*.

Tensile Force Caused by Longitudinal Restraint

Measurements were made of the longitudinal tensile force developed during testing standard-size specimens of steel *R* under a few widely different conditions. In all tests the tensile force rose to a maximum value very soon after the start, remained almost constant at this value for about half the duration of the test, and then decreased slightly.

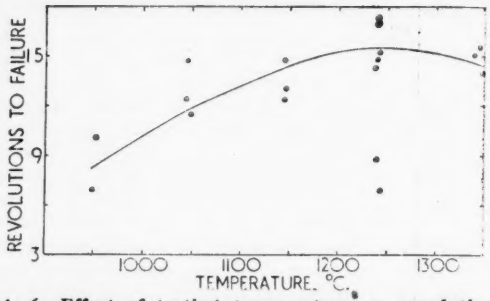


Fig. 6—Effect of testing temperature on revolutions to failure in torsion for test pieces cut transversely from a mild-steel billet (testing speed 100 r.p.m.)

The results given in Table II refer to the maximum value of the tensile force in each case.

Temperature Rise During Test

Table III gives the results of temperature-rise measurements carried out on mild-steel test pieces (not the same cast as steel *R*) which had a $\frac{1}{8}$-in. dia. central hole, enabling a thermocouple to be placed inside the test length. For comparison purposes, the temperature rise measured with the thermocouple in the usual position on solid test pieces of steel *R* is also given ; this value is usually lower.

Samples from a Mild-Steel Billet

Where material is only available in billet form, the most economical way of preparing test pieces is to make them from transverse slices. A curve showing the effect of temperature on the number of revolutions to failure for such test pieces is shown in Fig. 6, and is similar in form to the corresponding curve for the rod test pieces in steel *R* (Fig. 3*a*). The number of revolutions to failure is very much lower at all temperatures than for the rod test pieces, however, and the reproducibility is not so good. For comparison purposes, several tests were carried out at 1250° C. on specimens cut longitudinally from the same billet. The numbers of revolutions to failure were 63, 58, and 48 for test pieces cut from the outside of the billet, and 41, 41, and 43 for those cut from the centre.

DISCUSSION OF RESULTS

Interpretation of these results is complicated by a number of factors ; the three most important are the complex stress system caused by the superposition of

Table III

TEMPERATURE RISE DURING TORSION TESTS

Testing Temperature, °C.	Testing Speed, r.p.m.	Hollow Specimens with Internal Couple			Solid Specimens (Steel *R* with External Couple	
		Specimen	Temperature Rise, °C.	Revolutions to Failure	Temperature Rise, °C.	Revolutions to Failure
950	100	*S*4	40	19	25	26
1050	100	*S*2	Couple broke	25	22	35
1050	100	*S*9	53	27	22	35
1250	100	*S*3	28	37	variable (17–28)	64
1350	100	*S*10	13	29	14	57

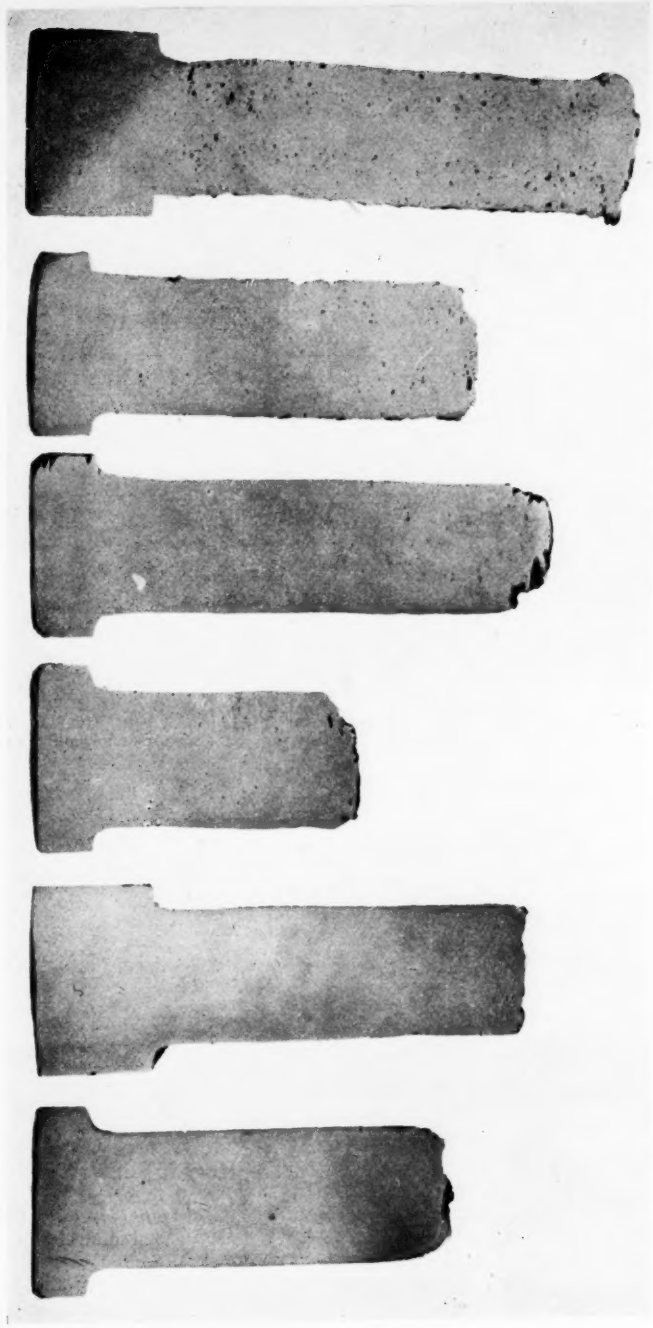


Fig. 7—Unetched longitudinal sections through broken torsion test pieces in steel *R*, showing varying degrees of cavitation for different testing speeds and numbers of revolutions to failure. Testing temperature 1350° C. × 2

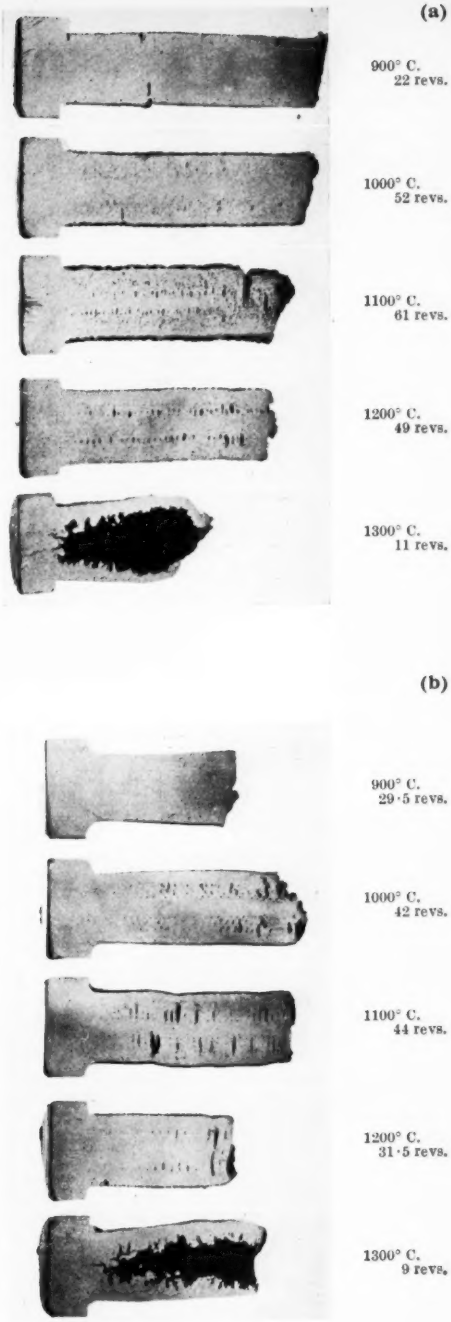


Fig. 8—Unetched longitudinal sections through broken torsion test pieces in steel *X* showing varying degrees of internal break-up for different testing temperatures and numbers of revolutions to failure. Testing speed : (*a*) 25 r.p.m. ; (*b*) 400 r.p.m. × 1

[*Hughes*

[*To face p.* 218

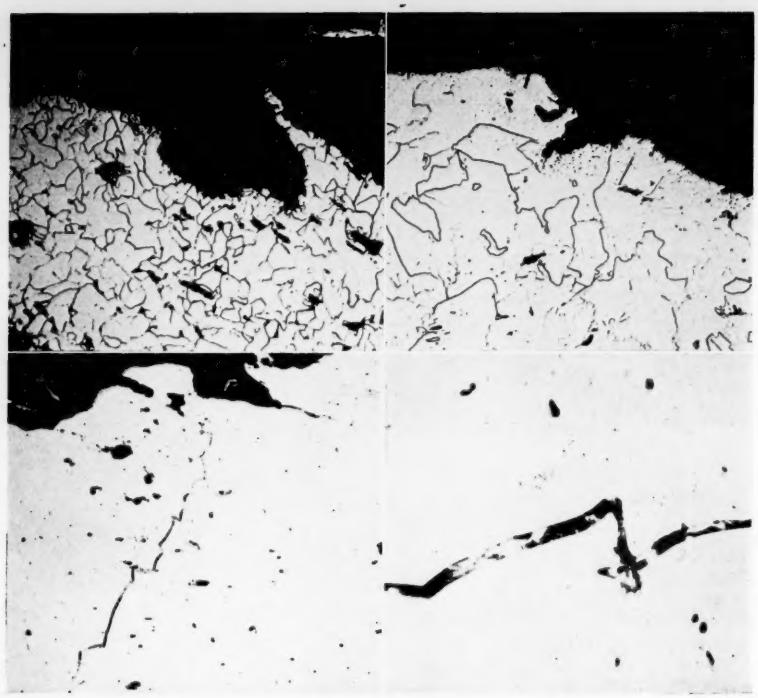


Fig. 9—Fractured zones tested at 400 r.p.m. : (*a*) Steel *R*, testing temperature 1250° C.; (*b*) steel *R*, testing temperature 1400° C. ; (*c*) steel *X*, testing temperature 1200° C. ; (*d*) part of same crack as 9*c* showing entrapped oxide

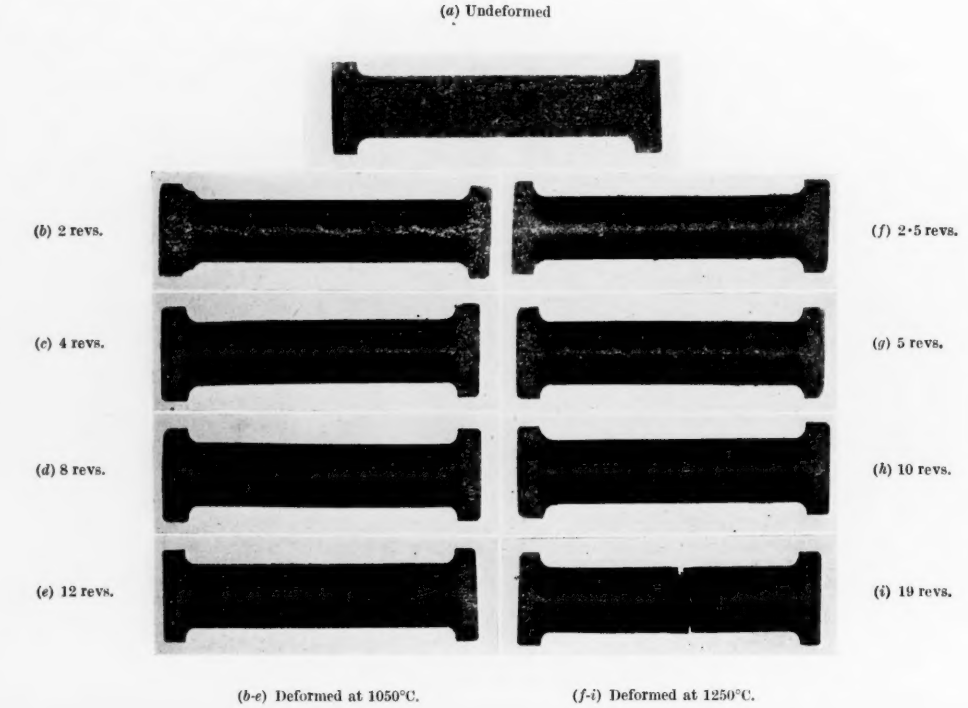


Fig. 10—Sulphur prints on longitudinal sections through partially deformed torsion test pieces in steel *FC*. Testing speed 100 r.p.m. × 1

Fig. 11—Unetched sections of test piece of steel *FC* deformed at 1250° C. : (*a*) Section from centre after 2·5 revs. ; (*b*) similar section after 19 revs. ; (*c*) section from outside after 2·5 revs. ; (*d*) similar section after 10 revs. ; (*e*) outside section after 19 revs. ; (*f*) similar section showing break associated with sulphide inclusions

tensile and shearing stresses, the internal break-up that often occurs before fracture, and the temperature rise that always occurs during the test. Any one of these factors may outweigh the others, according to the material and the testing conditions, and it is not possible to advance conclusive explanations.

Variation in Revolutions to Failure with Temperature

The temperature at which the maximum occurs in each of the curves (Figs. 3*a*, 3*b*, 5*c*, 5*d*, and 6), is not the temperature at which the metal is in the softest condition ; this effect has also been observed in hot-torsion tests on other materials. A few high-temperature tensile tests carried out on both steel *R* and steel *X* showed no decrease in reduction in area or elongation until the temperature of incipient fusion was reached. Moreover the torque/temperature curves show nothing corresponding to the maximum in revolutions to failure. This apparently typical form of curve, showing a maximum number of revolutions at a temperature below that of incipient fusion, is thought to be a result of the combination of tensile and shear stresses imposed on the test piece. The capacity of a metal for deformation and its resistance to deformation both vary with temperature, and together these properties, at a certain temperature and under the action of a complex stress system, apparently cause the maximum.

The curves for steel *R* (Fig. 3*a*) show that below about 1220° C. increasing testing speed causes increasing deformation, whereas above this temperature the opposite is the case. This suggests that two factors are affecting the results.

Below 1220° C. the often considerable rise in temperature during the test probably causes the falling torque characteristic. This temperature rise is of an order that would explain the effect of testing speed at 950° C., 1050° C., and, to a lesser extent, 1150° C., if the decrease in torque during the test is assumed to be caused by the temperature rise alone. No satisfactory explanation has been found for the reversed effect of testing speed at temperatures above 1220° C. ; when the steel is very soft the amount of deformation it can withstand is apparently diminished as the severity of the testing conditions increases.

Although the observed temperature rises for steel *X* and mild-steel *R* were similar under comparable testing conditions, the marked internal break-up, which occurred in nearly all test pieces of steel *X*, was probably the main factor causing the downward displacement of the curves of Fig. 3*b* with increasing testing speed. Once this break-up started, the prevailing stress conditions would probably determine the duration of deformation before fracture. In other words, the higher the testing speed the sooner, in terms of deformation, would failure occur. The maximum numbers of revolutions to failure for steel *X* were not on the whole a great deal lower than for steel *R*, with the exception of the tests on the latter at 12 and 50 r.p.m. Curves of the type obtained on steel *X* are obviously better for standardizing testing conditions than those of the mild-steel type. This variation in the position of the maximum with testing speed may be peculiar to mild steel and closely related materials, since tests on an austenitic stainless steel gave curves very similar to those for steel *X*.

For constant speed of rotation, the rate of deformation increases with test-piece diameter. At a testing speed of 50 r.p.m., the $\frac{1}{2}$-in. dia. test pieces show a maximum in revolutions to failure at about 1250° C. (Fig. 5*c*), the $\frac{3}{8}$-in. test pieces at 1350° C. (Fig. 3*a*) and the $\frac{1}{4}$-in. test pieces at a temperature probably above 1350° C. (Fig. 5*d*). The curves obtained for the $\frac{3}{8}$-in. test pieces show that the temperature at which the maximum occurs decreases with increasing testing speed, and this agrees with the observed effect of change in test-piece diameter.

Mode of Deformation and Fracture

The mechanism of deformation in torsion has two interesting features—the very large amount of deformation undergone by the outer parts of the test piece, and the transverse flow effect which seems to develop after appreciable deformation. The internal breaks which occur before final fracture are nearly always transversely elongated, and generally occur in two bands on either side of the central axis ; this effect was very marked with steel *X* (Fig. 8). The form and location of these breaks are probably connected with the stress distribution in the test piece. According to Nadai,[12] the stress distribution over the section of a test piece subjected to combined torsion and tension is of the form shown in Fig. 12,

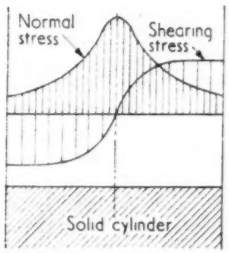


Fig. 12—Plastic stress distribution in solid cylinder under combined torsion and tension (Nadai)

the tensile stress being a maximum at the centre where the shearing stress is zero. Directional effects at right angles to the line of action of the tensile stress would probably facilitate the start of breaks, because of stress-concentration effects. The tensile stress, although greatest at the centre line, acts parallel to the comparatively undisturbed flow lines in the rod from which the test piece was made, and is consequently unlikely to start breaks here. At the outside of the test piece the tensile stress has a minimum value ; thus, although there may be a transverse flow effect, it is not likely to initiate breaks. Between the outside and the centre, however, the tensile stress is appreciable, and therefore any transverse directional effects that exist here may largely contribute to the initiation of breaks. It is significant that test pieces cut transversely from a mild-steel billet gave lower values for revolutions-to-failure than test pieces cut longitudinally from the same billet.

Further indications of the importance of transverse directional effects were provided by special tests on

steel *R*. A few test pieces, which had been given certain fractions of the amount of deformation required to cause failure and then cooled down to room temperature, were reheated and tested to failure under the same conditions. The number of revolutions to failure on retesting was always less than the number obtained in tests which had been carried to failure in one stage. The difference corresponded approximately to the amount of deformation imparted initially. In most cases the initial deformation was insufficient to cause any visible trace of internal break-up.

It has not been possible to determine exactly how the internal break-up starts. There was some evidence for the high-sulphur steel of association with transversely elongated inclusions, but there was none for steel *R* or steel *X*, which were both fairly low in non-metallic matter, and whose inclusions, at all positions away from the central axis, were broken up into extremely fine particles by the deformation.

Variation in Torque with Temperature

The relationships between torque and temperature (Figs. 5*a* and *b*) show no unexpected features. The curves are displaced to higher torque levels by increasing speed—a well-known property of metals at high temperatures. The torque figures for steel *X* are rather higher than for mild steel under comparable conditions, but the difference decreases with increasing temperature, and above 1100° C. is very small. If the torque values at a given temperature are plotted against testing speed in r.p.m. on logarithmic axes, they lie on a straight line. The dimensions of the deformed part of the test piece do not change appreciably during a torsion test, and so a constant speed of rotation gives a constant rate of deformation of the outer parts of the test piece. Thus, the relation between resistance to deformation and rate of deformation can be determined more easily than from tension or compression tests. The stress distribution across the section in torsion is not uniform but, if it is assumed that the shear stress remains substantially constant in zones which are undergoing plastic deformation (Fig. 12), curves connecting shear stress with rate of shear may be obtained.

APPLICATIONS OF THE HOT-TORSION TEST

The results obtained on both steels (except at very low testing speeds in the case of steel *R*) show that the maximum number of revolutions occurs in each case at the temperature which has been found in practice to be best for the rotary piercing of steel billets in the manufacture of seamless tubes. It is not possible to assess the relation of hot-torsion test results with practical experience in other hot-working operations—many of which are less severe than rotary piercing, and could probably be carried out at rather lower temperatures. The optimum temperature shown by the test would apparently always be a satisfactory temperature to use, and it would often be the most suitable. Possible applications of the test may be summarized as follows:

(i) To show the effect of composition and constitution on hot-workability.

(ii) To indicate what is likely to be the best hot-working temperature for a steel, and how critical this temperature is.

(iii) To compare the hot-workability of an unfamiliar steel with one whose behaviour is already known, by comparing the maximum numbers of revolutions to failure.

(iv) To compare the forces required to deform different materials at various temperatures, and thus to indicate how the power requirements of the mill or press are likely to be affected by a change of steel.

(v) For tests on batches of steel that appear to have anomalous hot-working properties.

Up to the present the author has had experience of the test only as a research tool, and although applications (i)–(iv) above are suggested with confidence, a full assessment cannot be made of application (v) or the possible use of the test for inspection purposes. The test can give no information about the effect of surface defects on the hot-working properties of billets, and many hot-working difficulties are due to defects of this kind.

Standardized testing conditions have been established for carbon and low-alloy steels, using a testing speed of 100 r.p.m. and test pieces machined either from small-diameter rod or transverse billet sections. Rod is preferred for research on the inherent working properties of steel; useful results can be obtained with billet sections but the reproducibility will probably not be so good.

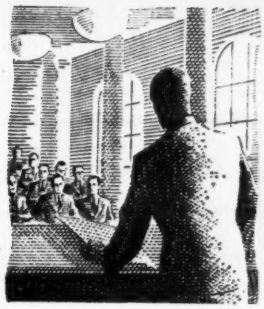
Acknowledgements

The author wishes to thank Messrs. Tube Investments Ltd. for permission to publish this paper; Dr. J. W. Jenkin, who suggested the investigation, for his interest and encouragement; Dr. C. E. Homer, Superintendent of Metallurgical Research, under whose guidance the work has been carried out, for advice and suggestions on a number of points; and Professor L. Aitchison for several helpful discussions.

References

1. A. Portevin, E. Pretet, and J. de Lacombe: *Rev. de Mét.*, 1936, vol. 33, No. 2, pp. 114–132.
2. A. Portevin and P. G. Bastien: *J. Inst. Met.*, 1936, vol. 59, pp. 83–106.
3. O. W. Ellis: *Iron Steel Inst. Carnegie Scholarship Memoirs*, 1924, vol. 13, pp. 47–81.
4. O. W. Ellis: *Ibid.*, 1926, vol. 15, pp. 195–215.
5. O. W. Ellis: *Trans. Amer. Soc. Steel Treat.*, 1933, vol. 21, pp. 673–699.
6. W. L. Kent: *J. Inst. Met.*, 1928, No. 1, pp. 209–226.
7. A. Morris: *Trans. Amer. Inst. Min. Met. Eng.* (Inst. Metals Div.), 1931, pp. 336–342.
8. M. Itihara: *Tôhoku Imperial University Technology Reports*, 1936–38, vol. 12, pp. 63–118.
9. C. L. Clark and J. Russ: *Metals Technology*, 1945, vol. 12, Tech. Publ. No. 1839, Dec.
10. H. K. Ihrig: *Ibid.*, Tech. Publ. 1932, Oct.
11. F. K. Bloom, W. C. Clarke, and P. A. Jennings: *Metal Progress*, 1951, vol. 59, pp. 250–256.
12. A. Nadai: "Plasticity," p. 216. New York, 1931: McGraw Hill Book Co., Inc.

DISCUSSION ON PAPERS



DISCUSSION AT MEETINGS · WRITTEN CONTRIBUTIONS

AUTUMN GENERAL MEETING, 1951

THE AUTUMN MEETING OF THE IRON AND STEEL INSTITUTE was held on Wednesday and Thursday, 21st and 22nd November, 1951, at the Offices of the Institute, 4 Grosvenor Gardens, London, S.W.1. The President, Mr. RICHARD MATHER, occupied the Chair for the greater part of the first day. During his absence his place was taken by Mr. K. HEADLAM-MORLEY, Secretary of the Institute.

Sessions were held from 10.0 A.M. to 1.0 P.M. and from 2.30 P.M. to 4.45 P.M. on the Wednesday, and from 10.0 A.M. to 1.20 P.M. on the Thursday. A buffet luncheon was provided in the Library of the Institute on the Wednesday.

The Report of the Meeting is published in this issue of the *Journal* (p. 187).

Discussion on the Paper—

PHYSICAL AND MECHANICAL PROPERTIES OF SEGREGATES IN TWO ALLOY STEELS*

By H. M. Finniston and T. D. Fearnehough

Dr. **H. M. Finniston** (Metallurgy Division, Atomic Energy Research Establishment) presented the paper.

Mr. **H. Allsop** (Brown Bayley's Steel Works, Ltd.): Finniston and Fearnehough conclude that the segregates are harder, less ductile, and give lower impact values than the matrix material. The hardness difference is demonstrated by the values given in Tables I and II, although it is not so apparent in terms of maximum stress. The elongation values, although generally lower on the segregate samples, are very erratic and, in some cases, are as good as those of the corresponding matrix material, particularly for samples tempered at the higher temperatures. Longitudinal impact tests (Table III), however, showed the segregate test pieces to give lower values than the matrix test pieces, but the difference is not very great and the former give surprisingly good figures. Could this result, at least partly, from the difference in cross-sectional dimensions of the tensile and impact test pieces? The tensile test piece had a gauge length diameter of 0·08 in. and the impact test piece a diameter of 0·12 in., although the depth behind the notch was only 0·084 in., and the impact test pieces may have been rather less completely segregate material than the tensile ones. Even if the former only included an annulus of matrix material about a core of segregates, the impact values could be expected to be higher than those of the segregate proper. If Figs. 3 and 4 are natural size, the inclusion of some non-segregate material seems to be a possibility. Furthermore, on freezing there is a gradual change in composition from matrix to segregate. In addition, the ratio of segregate to matrix (from Table VI) does not always show a marked degree of segregation. The results obtained on transverse impact tests (Table IV), in which the segregate lay in the path of fracture, confirm that longitudinal impacts may not have been solely segregate material since, when the segregate is effectively located behind the notch, the difference in properties is more clearly shown.

The authors state in conclusion (5) that metal in the interdendritic arms shows variations in hardness and composition comparable with those shown by the segregates. It follows therefore, that, because steel freezes over a range of temperature and thus its normal macrostructure consists of dendrite axes and arms and infilling metal, in commercially sound material, *i.e.*, material not segregated to the extent of the discard examined by the authors, segregate and matrix material should show differences in properties, etc., similar to those revealed by the larger segregates examined by the authors. This dendrite and infilling metal effect constitutes the normal banding or grain flow of wrought material.

The differences between dendrite and infilling material exhibited by macro-etching normal wrought specimens mainly of nickel–chromium steel of En 25 type, have been examined in our laboratories mainly in terms of hardness, and the toughness or ductility deduced from their behaviour during fracture. We have used the term 'segregate' to refer to the infilling material, *i.e.*, the

* *J. Iron Steel Inst.*, 1951, vol. 169, Sept., pp. 5–12.

Table A

MECHANICAL PROPERTIES OF TRANSVERSE TENSILE TEST PIECES

(Oil-hardened 850° C., tempered 620° C., 30 min., air cooled)

Cast No.	Position	Maximum Stress, tons/sq. in.	Elongation, %	Redn. in Area, %	Dendrite Hardness			Segregate Hardness			Difference
					Min.	Max.	Average	Min.	Max.	Average	
10930	Outside	67·0	13·0	29·1	310	317	316½	317	332	324	7½
	Midway	65·6	11·5	27·6	299	321	308	317	325	321	13
	Centre	66·2	11·5	26·1	305	309	308	319	331	321	13
E6472	Outside	65·2	15·0	33·5	299	303	301½	299	310	307	5½
	Midway	64·6	12·0	29·1	293	296	293½	303	317	308½	15
	Centre	63·0	9·0	26·1	286	293	289	303	313	307½	18½

less pure material, in a normal dendritic structure and not to segregates comparable in magnitude with those examined by the authors. Our experiments have shown that the segregate portions of a macrostructure are harder than the infilling material. The normal ' coring ' effect, which occurs in metals solidifying over a range of temperature, is the cause of the dendritic pattern which, after hot working, gives the banding or grain-flow effect. It might be expected, therefore, that the coring would be at a minimum at the edge of an ingot which solidifies quickly, and would become more pronounced towards the centre as solidification is slowed down, and that this difference would be preserved in the wrought section. We find that the dendrite-segregate hardness differences do vary in this way, the interdendritic hardness difference being small near the surface of the section and increasing towards the centre. Transverse tensile test pieces tend to give lower elongation values as their position approaches the centre of the section. These effects are represented by the values given in Table *A*.

Our work also confirms the authors' conclusions that in hardened and tempered material, irrespective of the tempering temperature, the segregate material maintains a higher hardness level than does the matrix, but our results suggest that the hardness difference increases with increasing tempering temperature, indicating that the segregates are more resistant to tempering. The figures in Table *B* show the increase in interdendritic hardness with increasing tempering temperature.

Our examination of the ductility of the segregate bands has been confined mainly to transverse tensile test pieces. A difference was invariably found between the hardness of the dendrite and that of the infilling material, and the maximum stress of the aggregate test piece corresponds more nearly with the nominal maximum stress (based on hardness tests) of the softer dendrite than with the harder infilling material as illustrated by the results shown in Table *C*, in which the tensile test value is the actual maximum stress obtained on a tensile test, corrected to the nearest whole ton.

Examination of the macrostructure of such broken test pieces, especially those giving low ductility figures, shows that the fracture very largely lies in the segregate or infilling bands, Figs. *A* and *B*, and not in the dendrite bands, which are softer. In more ductile pieces the

Table B

INCREASE IN INTERDENDRITIC HARDNESS WITH TEMPERING TEMPERATURE (CAST 31288)

Oil-hardening Temp., °C.	Tempering Temp., °C.	Maximum Stress, tons/sq. in.	Elongation, %	Reduction in Area, %	Dendrite Hardness			Segregate Hardness			Difference
					Min.	Max.	Average	Min.	Max.	Average	
800	200	100·8	4·0	5·0	454	460	457	458	471	465	8
	300	96·4	5·0	7·0	429	438	434	439	460	444	10
	400	90·4	6·0	13·4	415	427	421	435	442	438	17
	500	77·2	7·0	12·0	376	386	382	386	412	397	15
	600	72·4	11·0	21·5	341	364	351	360	376	367	16
	650	64·8	12·0	19·9	303	313	306	313	329	323	17
850	200	102·0	3·5	5·0	460	476	469	463	487	481	12
	300	100·4	6·5	6·7	450	466	459	460	483	470	11
	400	90·4	6·5	11·7	412	429	419	429	454	434	15
	500	85·6	6·0	8·4	383	410	395	401	435	413	18
	600	80·4	9·5	19·9	362	386	376	394	412	401	25
	650	67·2	10·5	23·0	306	319	312	319	345	330	18
950	200	102·8	4·0	6·7	460	469	466	470	480	474	8
	300	99·4	5·5	8·4	438	460	447	451	469	459	12
	400	91·2	5·5	6·7	415	426	422	426	435	430	8
	500	86·0	6·5	10·1	383	399	398	410	426	422	24
	600	83·0	8·0	15·1	391	403	393	415	429	418	25
	650	70·6	7·5	13·4	321	343	331	345	376	362	31

Table C

RELATION BETWEEN HARDNESS AND MAXIMUM STRESS

Cast No.	Forging No.	Average Hardness		Calculated Max. Stress, tons/sq. in.		Experimental Maximum Stress of Aggregate, tons/sq. in.
		Dendrite	Segregate	Dendrite	Segregate	
31351	A2	225	254	48	54	49
	A9	226	252	48	54	48
	A13	218	250	46	53	48
31352	B6	248	267	53	57	53
	A7	246	256	53	55	52
	A10	240	261	51	56	52

fracture path is confused because of the local distortion of the structure resulting from the high elongation and reduction in area values. Transverse bend tests on samples previously etched on the side in tension, also indicate that cracking occurs preferentially in the segregate bands. Thus, in a composite material of two hardness levels, fracture takes place preferentially in the harder one, but at approximately the maximum stress of the softer one.

The failure of the segregate material at a lower load than that expected from its hardness is indicative of premature failure, the most likely cause of which is brittleness. Figure *C* shows a macro-etched section up to the fracture of a longitudinal test piece from a bar of about 6 in. dia. The rupturing of the segregate bands is analagous to the well-known 'cupping' in segregated cold-drawn wire. A tensile test piece initially elongates along its full gauge length, but after fracture the elongation varies from a maximum per unit length at fracture to a minimum at the end of the gauge length and, since in both transverse and longitudinal test pieces the secondary fractures occur within a comparatively short distance of the main fracture, it is evident that the segregates are capable of some deformation and are therefore relatively brittle. This observation again confirms the findings of the authors.

In our work many specimens from different casts, cast into the same sized ingots and made into the same sized forgings, were examined. Transverse tensile and impact tests frequently showed quite marked differences in the ductility between different casts but we were unable to detect corresponding differences either in macro-structure, interdendritic hardness variation, or number and rating of inclusions. I think that the authors give more weight to the effect of non-metallic inclusions on the behaviour of the segregates than is merited. The variations in composition reported by the authors do not satisfactorily explain the apparent difference in behaviour between dendrite and segregate material.

The performance of material in respect of transverse mechanical properties depends on the characteristics of the segregate bands and is primarily a cast property, and may depend on the steelmaking technique employed.

The causes of the variation in properties of the segregate bands are very important, since they form an integral part of the normal macrostructure of all wrought objects and their properties may determine the success or failure of parts subject to transverse or circumferential stress, either in testing or in service.

Dr. **W. Betteridge** (Mond Nickel Co., Ltd.): The authors have succeeded in determining the chemical nature of segregates observed in two alloy steels and the differing mechanical properties of such segregates when the body of steel is heat-treated, and it is of interest to consider the reason for the detected changes in properties. The two main factors controlling the differences in mechanical properties of matrix and segregate are the differing chemical composition, which results in the segregate having different transformation characteristics from the matrix, and the higher non-metallic inclusion content of the segregate. Of the mechanical properties measured, hardness is unique in being unlikely to be significantly affected by the inclusion content, so that it appears reasonable to accept it as a measure of the changes in properties resulting solely from the differing composition, and therefore the differing hardenability, of the segregate.

If the other properties measured after differing tempering treatments—the ultimate tensile stress, the elongation, and the impact strength—are plotted against the hardness values, mean curves can be obtained both

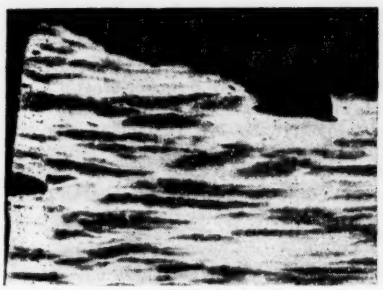


Fig. A

Fig. B

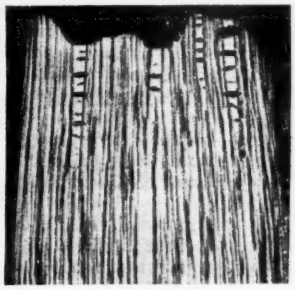


Fig. C

Figs. A and B—Macrostructure of broken transverse tensile test pieces

Fig. C—Macro-etched section up to the fracture of a longitudinal test piece

for the matrices and for the segregates. If the change in properties were associated only with the different degree of transformation, the two curves should be coincident, since the authors state that the microstructures of the two types of material after hardening and tempering show little difference. In practice it is found that the curves for ultimate stress and for elongation are displaced in such a way that, for a given hardness level, the segregate has a lower ultimate stress and lower elongation than the matrix. It is concluded that these displacements result from the increased inclusion content. No significant displacement is to be noticed, however, for the impact values, and it would appear that the changes in impact strength between matrix and segregate after a constant treatment arise solely from the difference in hardenability.

Micro-radiographic work by various authors, published since the work described in this paper was carried out, has indicated that chromium is particularly liable to segregate and, because of its effect on hardenability, it should have a marked influence on the mechanical properties of segregates. It is therefore unfortunate that the results published in this paper are confined to steels which are chromium-free.

Dr. **L. Northcott** (Armament Research Establishment) : Figures 5–8 show that the materials which segregate are principally nickel and manganese : the authors have adopted the same scale of one division per 0·1% difference in composition, and the nickel content ranges from 3·6 to 3·8%—a two-division difference. The variation in chromium content is much less (0·2% to a fraction over 0·2%). In their conclusion (3) on p. 12 the authors say : "There is a composition gradient across each segregate, rendering them more highly alloyed in nickel, vanadium" and so on, and they add, "There is also a general increase in carbon, sulphur, and phosphorus content." The figures given in Table VI, showing sulphur and phosphorus contents exceeding 0·1%, suggest, however, that these are far more damaging to the material than an increase in nickel content from 3·4 to 3·6%, and similarly with the other alloying elements.

In Table I, with a 300° C. tempering temperature the mean figure for the maximum stress of the segregate is lower than the mean figure for the maximum stress of the matrix, although the hardness is higher. I think that if the three specimens that broke had been tested in a truly axial manner—which is extremely difficult with any size of test piece, and particularly with small ones—probably rather higher figures would have been obtained. The figures that the authors quote for the maximum stresses where a proof stress is also given tend to support this view.

The authors explain the fact that the micrographs indicate that the segregate is lower in carbon than the matrix by the increased phosphorus content gradient, but the actual compositions are rather higher in carbon in the segregate than they are in the matrix. Are the carbon figures quite up to the standard of the others ?

Mr. D. R. Thorneycroft (Mond Nickel Co., Ltd.) : I believe that massive segregates are to be found generally in exceedingly large ingots and forgings and occur less frequently, if at all, in small ingots or castings. The authors have confined their attention to the massive segregates, but they also state that the surrounding dendritic material, which constitutes the matrix, as illustrated in Fig. 11, may, on the basis of spectrographic tests, show similar variations in alloy content between the dendritic spines and the interdendritic region.

I have recently examined several 1¼-ton ingots of B.S. En 24 steel to determine the pattern and extent of the variation in transformation characteristics at different positions in the ingot. These ingots exhibited the well-defined columnar and equiaxed zones of macrostructure, which consisted entirely of the fine dendritic structure of which the large forgings examined by the authors are mainly composed. The individual dendrites were found to display a common pattern of transformation in which reactions were initiated in the dendritic spines of the structure and then extended progressively to the interdendritic regions. This sequence of structural change is indicative of a chemical composition gradient between the spines of the dendrite and the interdendritic region.

Furthermore, the M_s temperatures of the dendritic and interdendritic regions were different, and the differences could be accounted for by a difference of about 0·2% of carbon or by a difference in total alloy content (*i.e.*, Si + Mn + Cr + Ni + Mo) of about 3% between the two extremes of dendritic structure. The authors' analyses in Tables V and VI reveal that the massive segregates were on the average about 0·5% richer in alloying elements than the matrices, and the microchemical analyses show the increase in carbon content in the segregate to be not greater than 0·04%. It would seem likely that micro-segregation in a fine dendritic structure is also confined mainly to the alloying elements.

It would be of interest to examine massive segregates by the micro-radiographic method to determine whether the nickel segregates are as pronounced as the spectrographic and micro-chemical tests suggest. In the field of micro-radiography, tests on nickel-containing steels have not provided marked evidence of nickel segregation, whereas chromium and manganese segregation has been clearly defined. Micro-radiographic tests at selected positions in the cross-section of the 1¼-ton ingots of B.S. En 24 steel confirmed that the evidence of nickel or molybdenum segregation was slight.

Tensile test data, recently obtained on large-diameter forgings of 1½% Ni–Cr–Mo steel, which exhibited massive segregates towards the centre but not at the rim, showed that the segregates were harder than the surrounding matrix and were responsible for low ductility in the fully hardened and tempered material from the centre of the forgings. The loss in ductility was greater at a tensile strength of more than 80 tons/sq. in., and the segregates, although stronger than the surrounding matrix, were less ductile, and fracture occurred preferentially through their brittle structure. This effect was reduced by raising the tempering temperature, which induced greater ductility in the segregate, so that fracture occurred generally in the weaker matrix.

The authors' longitudinal impact tests on segregates and matrices, given in Table III, have been made on oil-hardened material of the two alloy steels. The heat-treated blocks from which the test pieces were machined were presumably the same size as the blocks used for the hardness tests. It is probable that in this section the steels were not fully hardened in the matrix or segregate, particularly in the case of the 3½% nickel steel. The presence of transformation products, other than martensite, would account for the low impact values on the 3½% nickel steel at the lower tempering temperatures, but if the tests were repeated on the water-quenched sample the differences between the impact values of the segregates and the matrices would probably be less pronounced.

CORRESPONDENCE

Mr. J. K. Hurwitz (Department of Mines, Canada) wrote : I would like to add a few comments on the interpretation of the microanalysis of the segregates.

Microanalytical equipment, based on the design of Convey and Oldfield,* has been built and tested at the Mines Branch to assist in the analytical work associated with diffusion as well as with segregate and inclusion problems in metals and alloys. Figure 6 of the paper by Convey and Oldfield illustrates that a traverse across a boundary with an infinite concentration gradient gave traverse curves of the **S** type. Hence, an apparent smearing out or spreading of a concentration gradient may be expected at the boundary of a segregate or inclusion. Again, if the width of the segregate were narrower than the sparked area, the spark would never be wholly within the segregate, and the true analysis would not be directly determined. It appears, therefore, that the technique had definite limitations. A detailed mathematical analysis was made of the expected exposure at each point on the photographic plate during a traverse of a boundary formed by two steel blocks of different composition, and good agreement was obtained with experimental data. Several parameters were found to be of importance, *i.e.*, the dimensions and shape of the sparked volume, the lengths of the spectrograph and microphotometer slits, and the ratio of photographic plate speed to sample speed. The most important parameter was the sparked volume. (Since it was necessary to consider the depth of sparking it is more correct to speak of sparked volume rather than of sparked area.) Several different shapes were assumed for the sparked volume, *i.e.*, a flat disc of uniform depth, a cap of a sphere, and a paraboloid of revolution. The flat disc held true for small speed ratios and the other two gave better agreement for large speed ratios (10 : 1 and higher ratios), since the spark remained longer on one position and vapourized a greater volume of sample.

The analysis of a segregate, such as those considered by the authors, involves the solution of a double boundary problem from a theoretical point of view. Such a calculation has been performed : the true peak concentration may be determined directly only if the width of the segregate is equal to or larger than the width of the sparked volume at the surface of sample plus the length of the spectrograph slit.

With narrow segregates, the true peak concentration may be estimated if the width of the segregate is known. The apparent width of the segregate will always be larger than the true width by an amount which is a simple function of the parameters already mentioned. With reference to Figs. 5–8, the following conclusions may be drawn : The true peak concentration probably has been obtained since the segregates are wider than the reported width of the sparked volume at the surface. The apparent width of segregate (obtained from the traverse results) always seems to be wider than the true width (shown in the macro-pictures) by an amount which is in good agreement with the above theoretical results. The results of the calculations will be published shortly.

AUTHOR'S REPLY

Mr. **T. D. Fearnehough** (Bragg Laboratory) wrote : Mr. Allsop's suggestion that our segregate test pieces were not pure segregate is concurred in, but we believe the amount of actual matrix included to be proportionally small. Figure *D* shows the test pieces of Fig. 12 of the paper etched to reveal the segregate position. The non-homogeneous nature of the segregates themselves must have considerable influence upon their mechanical properties. In the segregate impact test pieces the notch would eliminate the effect of any outer layer of matrix material.

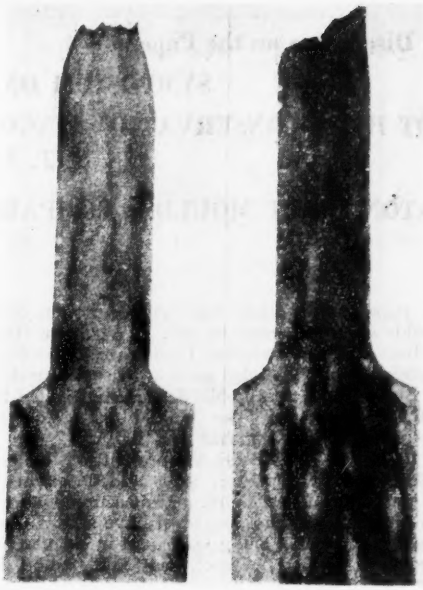


Fig. D—Test pieces etched to reveal the segregate portion

Mr. Allsop's conclusion that there is a strong tendency for fracture to occur preferentially through segregate material is most important. We believe that the association of inclusions with massive segregates also influences breaking strength, especially where inclusions break the surface and act as stress raisers.

Dr. Betteridge has pointed out that hardness tests are not influenced by inclusion content and, by plotting hardness against ultimate tensile strength, elongation, and impact strength, has shown the possible effect of inclusion content on these properties.

Dr. Northcott's remarks on the segregation of various elements are concurred in. It is agreed that the high sulphur and phosphorus contents are likely to be most damaging to the segregate material, but it is the segregation of alloying elements that has most effect on differential hardenability. In regard to carbon content, we feel satisfied that the average carbon content of the segregate material is higher than that of the matrix, but the carbon appears to vary considerably within the segregate and there are definite indications that carbon tends to be highest at the segregate boundaries. Distribution of carbon in segregates is greatly influenced by phosphorus distribution.

Mr. Thorneycroft's remarks on the examination of $1\frac{1}{4}$-ton ingots indicate similarity between massive segregates and interdendritic filling. The further information of preferential fractures through segregate material is noted. The possibility of reducing the effect of segregates by suitable heat-treatment is of great importance.

Referring to Mr. Hurwitz's remarks on the spectrographic analysis of segregates, the traversing technique was developed by Convey and Oldfield specially for segregate analysis. We realized that the technique was limited by the spark volume, but the segregates examined were of sufficient cross-section to give a true peak analysis.

* J. Convey and J. H. Oldfield, *J. Iron Steel Inst.*, 1945, No. II. pp. 473P–507P.

Joint Discussion on the Papers—

SYMPOSIUM ON STRESSES IN MOULDS*

INGOT HEAT CONSERVATION: INGOT MOULD TEMPERATURE MEASUREMENTS†

By R. T. Fowler and J. Stringer

TEN-TON INGOT MOULDS: COMPARISON OF DESIGN AND CONDITIONS OF USE‡

By A. Jackson

The papers comprising the Symposium on Stresses in Moulds were presented by Mr. **J. Woolman** (Brown-Firth Research Laboratories, Chairman of the Stresses in Moulds Panel), who also gave a general introduction to the Symposium, and by Mr. **J. W. Grant** (B.C.I.R.A.), Mr. **W. C. Heselwood** (The United Steel Companies, Ltd.) and Mr. **W. H. Glaisher** (B.I.S.R.A.).

Dr. **R. T. Fowler** (B.I.S.R.A.), presented the paper by himself and Mr. Stringer, and added: Since this paper was published, weights and measurements of test ingots of killed and rimming steel have shown that the assumed density of 7·856 g./c.c., used in calculating the heat content in cal./g. of steel, was too high, and figures of 7·3 and 6·67 g./c.c. respectively were more appropriate. Consequently, the data given in Figs. 13 and 14 should be corrected by a factor of 1·09 for trial 4 and by 1·18 for trials 5 and 6. The data refer only to a thin horizontal section at mid-height of the ingot and mould and it is assumed that heat transfer normal to this section is negligible.

The measured surface temperatures have been used in a stepwise calculation of the temperature gradients through the mould wall, using a method developed § from a suggestion made by Professor Sarjant and others. Allowing for the actual mean density of the steel, agreement between experiment and calculation was good. A subsequent publication‖ gives further information on the variation of heat content of ingot and mould experienced in practice.

Mr. **A. Jackson** then presented his paper.

Mr. **N. H. Bacon** (Messrs. Steel, Peech and Tozer): The problem of mould design is very complex because the heat absorbed by the mould at each filling is distributed in an irregular manner, as shown by Fowler and Stringer. Although these authors aimed at determining the heat loss from an ingot, their paper also gives an indication of the heat absorbed by the mould and how it is affected by the surroundings, *i.e.*, by the pit walls, by full or empty moulds, and by slag pots. It is obvious that casting pits should be of sufficient size to avoid such great irregularities in mould temperature.

Some means of cooling, *e.g.*, by fans, might improve the mould life. In trial No. 4, for example, 60 min. after casting, the narrow side of the mould, which was next to an empty mould, was 60–90° C. cooler than the opposite narrow side, which was adjacent to a slag pot (*see* p. 287). The trials also showed that an adjacent full mould led to higher temperatures than did the close proximity of a slag pot. The effect of the pit walls appears to be similar to that of adjacent hot moulds.

The normal temperature, 125° C., at which the moulds were used seems to be very high; even half-way up there were variations of 90° C. from one part of the mould to another. The authors show that the corners of the mould may sometimes get hotter than some of the faces, which may explain why some companies make the corners as thick as the walls.

Mr. Jackson largely overcame the irregular heating of the mould by designing his bottom plates so that the moulds were regularly spaced. In obtaining a mould life of about 200 and a consumption of 11 lb./ton, for 10-ton slab moulds, Mr. Jackson has probably achieved a world's record. His work, in addition to demonstrating the importance of removing the mould from the ingot at the earliest possible time compatible with good steelmaking practice, has proved that moulds with an initial temperature of about 50° C. suffer less damage during service than moulds that have cooled to room temperature.

The curve in Fig. *A*, which is based on the information in the author's paper, flattens out, indicating that after 120 min. any further increase in stripping time has very little influence on mould life, provided, of course, that sufficient time is allowed after stripping for the mould to cool down to about 50° C. With a stripping time of 2–2½ hr., which is often necessary with brick-top moulds, it is still possible, therefore, to get a life of about 100 and a consumption figure of 20–22 lb. per ton of steel—a great improvement on the normal 50 lb. for brick-top moulds.

The work described in the Symposium was carried out to investigate the possibility of putting ingot-mould design on a more scientific or mathematical basis. Although this aim has not been realized, the work has removed doubts on the advisability of proceeding with the preparation of recommendations on mould design, which the Ingot Moulds Sub-Committee has long had in mind. Heselwood and Pickering indicate that irons having the higher growth rates may result in moulds

* *J. Iron Steel Inst.*, 1951, vol. 168, July, pp. 261–301.
† *Ibid.*, 1951, vol. 167, Mar., pp. 283–288.
‡ *Ibid.*, pp. 289–301.

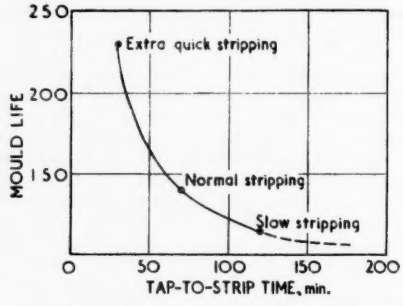


Fig. A—Relation between mould life and tap-to-strip time for 10-ton moulds

§ "Calculation of Ingot Temperatures from Mould Surface Temperatures," Paper PE/A/50/50: British Iron and Steel Research Association.

‖ "Cooling of Rimming Steel Ingots in a Casting Pit," Paper PE/A/39/51: British Iron and Steel Research Association.

giving shorter lives. Palmer* had previously shown that the growth characteristics of iron depended mainly on the silicon, manganese, and phosphorus contents, and that higher silicon induced greater growth and that higher manganese and phosphorus reduced the rate of growth. The results experienced in practice also indicate that growth and mould life are related. The irons investigated by Palmer were higher in silicon and phosphorus than ingot mould irons. Heselwood and Pickering examined samples cast at the same time as the ingot moulds ; their results are therefore of special value in connection with the study of mould performance.

From the authors' formula (p. 286) I have calculated the growth for a large number of moulds and have compared this with the mould life ; the results are shown in Fig. *B*. To obtain curve *a*, 429 moulds made in foundry *A* were used. Curve *b* refers to 245 moulds of the same pattern made in foundry *B*. Foundry *B* moulds gave a lower life and there is much greater scatter, suggesting that some other important factor, which is not present in foundry *A*, is operating in foundry *B*.

This growth characteristic appears to be important and worth pursuing. Rocquet and Olette† have given a useful practical method of making growth tests on a works basis, with sufficient accuracy.

Monsieur **M. Olette** (Institut de Recherches de la Sidérurgie) : Mr. Bacon has referred to a recently published report, in which M. Rocquet and I tried to define the part played by the different factors affecting cast-iron growth, within the range of temperature usually found in ingot moulds. A series of cast-iron bars, taken from one melt, was subjected to a number of cycles of heating up to a chosen temperature and cooling down to 400° C., first in a vacuum and then in an oxidizing atmosphere. The iron had the following average

* S. W. Palmer, *Journal of Research and Development*, 1947, vol. 1, pp. 361–378.

† P. Rocquet and M. Olette, *Fonderie*, 1951, vol. 67, pp. 2565–2577.

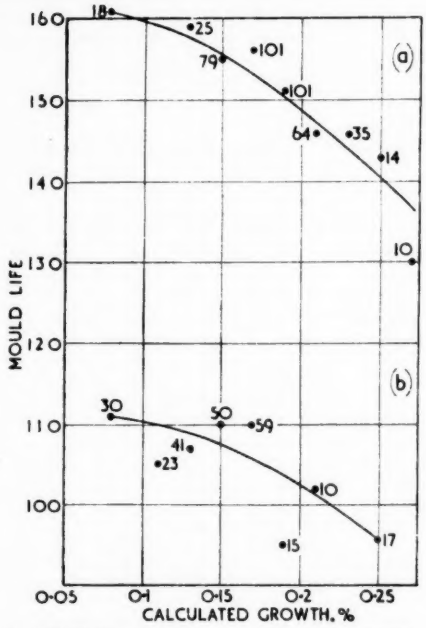


Fig. B—Relation between growth and mould life : (*a*) Foundry *A*, *n* = 429 ; (*b*) foundry *B*, *n* = 245

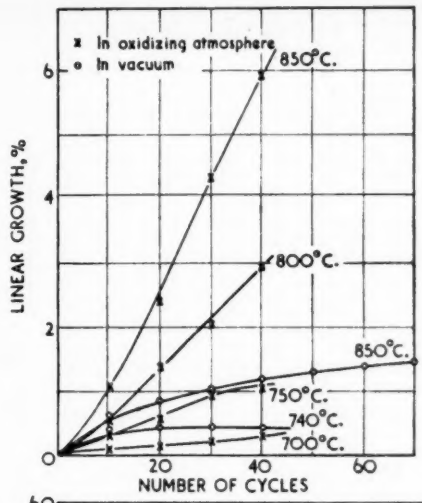


Fig. C

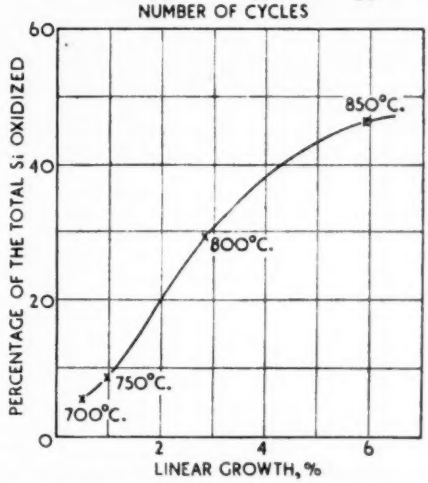


Fig. D

Fig. C—Relation between linear percentage growth of test pieces and number of cycles

Fig. D—Relation between oxidized silicon content and linear growth of each test piece, after 40 cycles in an oxidizing atmosphere

analysis : Total carbon 3·24%, graphite 2·70%, combined carbon 0·54%, silicon 1·62%, manganese 0·58%, phosphorus 0·12%, sulphur 0·08%.

Figure *C* gives the results of linear growth, obtained by measuring the length between two stops milled on the bars, before and after treatment. This marking removes the possibility of errors caused by a surface oxidation of the test piece, which occurs during treatment in an oxidizing atmosphere. The same method was used for treatment in a vacuum.

We did not use a dilatometric setting, as this needs many joints, the tightness of which is not always easy to ensure. Moreover, the circulation of an inert gas, free from any trace of oxygen, seemed rather difficult to realize. We therefore carried out tests in a high vacuum (1μ of Hg) in order to avoid the possibility of oxidation. More details of the experimental method are given in our paper.

The tests in vacuum showed that, below the transformation point Ac_1 (about 775° C. for the cast iron studied), and after about 20 alternate heating and cooling

cycles, the linear growth approached a limiting value of about 0·5%. A micrographic examination of the bars, before and after treatment, showed that pearlite and cementite were completely transformed into ferrite and graphite. These results agreed well with Heselwood and Pickering's results obtained after a 5-hr. isothermal treatment at 700° C. (*see* Fig. 31 of their paper), but above the transformation point the temperature cycles gave a larger growth, which increased slightly but steadily even after 60 cycles.

This result may be explained, as the authors suggest, by successive solution into the γ-phase and precipitation from the α-phase of small quantities of graphite, and also by distortion and dislocation of the micrographic components of cast iron at the transformation points Ac_1 and Ar_1, as mentioned by Benedicks and Löfquist as early as 1927.* The linear growth found after 70 cycles was much smaller than the growth noted by the authors with similar cast irons. This suggests that oxidation phenomena were not negligible in their experimental conditions.

Tests in an oxidizing atmosphere at 700° C. gave growth values smaller than those found at 740° C. in a vacuum. At this stage graphitization is probably not yet complete and the influence of oxidation is negligible. At 750° C., the results are higher than at 740° C. in a vacuum: here oxidation begins to have an influence. Above the transformation points, at 800° C. and 850° C., the growths are also very much larger than at 850° C. in vacuum, indicating the influence of oxidation phenomena on cast-iron growths.

Determination of the silica content in bars subjected to temperature cycles in oxidizing atmospheres, indicated the existence of a relation between the percentage growth and the percentage of the total silicon oxidized, as shown in Fig. *D*. Determination of the silica content in ingot moulds after use showed that an important part of the silicon is oxidized near the inner wall. Our results indicate that in the middle of the faces a 20% volume growth will take place on the inner surface of the ingot mould in a 10–15-mm. cast-iron layer 10 to 15 mm. thick. In the corners, where the temperatures reached are lower, this layer is thinner. This chemical corrosion may be responsible for the stresses appearing near the outer surface of the ingot mould; they would reach their maximum value in the middle of the faces. Strains from growth would then be additional to the expansion strains that appear during teeming.

Mr. **F. A. Gray** (The United Steel Companies Ltd.): My remarks will be mainly devoted to the paper by Fowler and Stringer. For maximum heat economy, we must know the minimum time in which an ingot can be stripped to permit safe handling, and also the minimum transfer time to the soakers. There is a point at which the heat content of the cooling ingot and that required for rolling are the same, and this ideal should be attained as nearly as possible. Thus, if we are to plan the procedure between teeming and stripping, and stripping and rolling, we must know the temperature and heat conditions within the ingot at any time. There are so many variables and basic data which are unknown that it is unlikely that a solution can be arrived at mathematically; the required knowledge must therefore be obtained largely from experimental measurements.

Fowler and Stringer have determined the heat flow from ingot to mould with a view to deducing the temperature and heat conditions in the ingot itself. The authors have successfully measured the temperature distribution within the mould at a number of points under very difficult conditions. They have been wise to use thermocouples inserted into the mould, because the measurement of surface temperature by the types of contact couples now in use is liable to large errors.

Do the authors intend to use the new hemispherical types of surface pyrometers, recently developed by B.I.S.R.A. and Land,† and to compare the results with those obtained by inserted thermocouples? If the new instruments are suitable and reliable the experimental technique will be greatly simplified.

Assessment of the conditions within an ingot by temperature measurement alone, whilst giving valuable information, is an indirect method, and the ideal would be to measure the temperature in the ingot itself. This, however, involves considerable practical difficulties.

Interest in the temperature and heat distribution within a cooling ingot is not confined to the fuel technologist but is equally important to the metallurgist and steelmaker. Mr. Jackson's paper shows that reduced stripping times have resulted in a marked increase in mould life. It would be interesting to hear from Mr. Jackson what effect these reduced stripping times, and therefore higher temperature ingots, have on the output and fuel consumption of the soaking pits.

Radiation and convection loss is given as only 10% of the heat gained by the mould; in other words, the mould takes up 90% of the heat flowing from the ingot. If this is so, ingot cooling would be accelerated by using thicker moulds. Mr. Jackson's new designs of both moulds and bottom plates, which gave considerably higher lives, had thicker walls.

Figure 14 of the paper by Fowler and Stringer, shows that the heat lost by the steel in 100 min. after teeming is about 90 cal./g. What temperature drop does this represent? A further point of interest which is brought out in this paper is the temperature rise of the casting-pit wall and the effect which this may have in raising the temperature of the ingot moulds prior to teeming.

The temperatures of the mould external surfaces, given in Fig. 8 of Jackson's paper, are considerably less than those given in Fig. 6 of the paper by Fowler and Stringer for very similar wall thicknesses. The latter authors found that the temperature of the broad face of the mould was higher than the temperature of the narrow face, whereas Jackson found that the narrow face was slightly hotter.

A joint investigation by the metallurgist and fuel technologist on the cooling of an ingot would be advantageous.

Mr. **H. Brooke Freeman** (John Summers and Sons, Ltd.): In the paper by Fowler and Stringer, a horizontal slice of the mould was made to serve as a calorimeter to measure the heat loss from the ingot at any moment. The results obtained indicate that the method is practical and reproducible. The few trials reported in the paper show how the temperature gradients in the mould walls vary with the external conditions. The paper deals with the development of a method for recording the heat losses from an ingot: further experiments will show whether the method will give results of practical value.

On p. 287, the temperature contours shown in the mould walls indicate differences of 150° to 250° C. between the inner and outer faces of the walls. Whereas the developed surface temperature contours may be considered reliable for the external surface of the mould, very steep temperature gradients may arise in the innermost layer of the mould, because it is in contact with molten steel. The temperatures given on the inner face of the moulds (Fig. 10) would then be incorrect. I am interested in this paper primarily from the metallurgical aspect and I want to know the rate of heat

* *J. Iron Steel Inst.*, 1927, No. I, pp. 603P–633P.

† M. D. Drury, K. P. Perry, and T. Land, *J. Iron Steel Inst.*, 1951, vol. 169, pp. 245–250.

extraction from the ingot in the first few seconds following teeming, because, during this time the skin of the ingot is formed, and the quality of that skin will be reflected in the surface condition of the finished product.

I am concerned particularly with 6½–8-ton low-carbon (about 0·07% C) rimming-steel ingots, cast in slab moulds. The rim of a rimming-steel ingot consists of 5 layers. The first consists of spattered nodules ejected by the steel as it is rising in the ingot. These small particles of steel are ejected by the gases and frozen on the mould walls. The second layer is quenched on to the first layer by the drastic cooling effect of the mould. The third layer, produced by the reaction of $FeO + C \rightarrow CO + Fe$, forms a very dense and solid skin. The fourth layer is that usually indicated by the rim holes, and the fifth that indicated by rim channels. The skin formation is of particular importance in making full-finished auto-body sheets, because these layers determine the quality of the surface of the sheet, unless very extensive de-seaming is carried out on either the ingots or the slabs.

It will often be found that the second layer is porous (the porosity being quite different from that caused by the rim holes) and the small pores formed lead to the formation of surface defects. Since the quenching action is very quick, the second layer is formed in less than a second. The temperatures in this layer in the period immediately following, when the pores are formed, are therefore of great interest.

A rimming-steel ingot teemed into a hot mould, behaves differently and has a very different surface condition from one teemed into a cold mould. Within narrower limits of mould temperature the differences tend to be masked by other factors. As the metal touches the mould walls there is instantaneous solidification, followed by a severe quench. The pick-up of heat by the inner face of the mould must be considerable, and the temperature that it reaches will depend primarily on the rate at which the heat is conducted away from the inside face. The rate of teeming, the condition of the metal—rimming action, and so on—will affect the conditions at the mould face, and will indirectly affect the formation of the ingot skin.

The method reported in this paper for measuring the heat loss from the ingot could be extended to record the temperature of the inner face of the mould. The plugs might even project sufficiently into the mould to record the temperatures obtained in, say, the first ¼ in. of the ingot skin. Information obtained in this way would aid us considerably in our conceptions of the mechanism of ingot skin formation, and might also throw considerable light on ingot cracking.

Jackson has shown that the lives of moulds are affected by the temperature cycle to which they are subjected and the method devised by Fowler and Stringer would give more precise information on temperature gradients and peaks in the mould walls and might lead to a more accurate appreciation of the conditions that determine mould life.

In doing this work, why did the authors depart from the practice of the originators of this method by using a mild-steel plug for the thermocouples, instead of a cast-iron plug ? Can they provide a plot of the developed-surface-temperature contours at the inside face of the mould, and, if so, within what limits of accuracy would these contours be valid.

Mr. **Neil H. Turner** (Appleby-Frodingham Steel Co.) : Although the method devised by Fowler and Stringer only permits an estimation of the amount of heat removed from the ingot, it has the advantage that, when suitable conditions can be provided, its use is likely to lead to less interference with the sequence of operations than a direct method. The secondary purpose of the paper may not be adequately fulfilled, because the temperature measurement is limited, as Dr. Fowler has said, to one horizontal cross-section of the mould.

The value of the paper to the instrument engineer could have been further enhanced had sufficient information been available to explain certain apparent peculiarities. For example, is the flattening of the curve in Fig. 6 between 35 and 45 min. necessarily caused by the effect of solidification ? The curve in Fig. 4 shows a similar trend at approximately the same time—it is not stated, however, whether this curve is representative of the general trend or of one particular case. A difference of approx. 75° C. exists between the middle of the terminal board and the end of it. An indication of the size of this board and its exact position in relation to the ingots would have permitted a clearer appreciation of the reason for this temperature difference. This would have avoided the question of whether the correction due to the heating of the terminal board causes an apparent rather than a real slowing down of the temperature rise of the ingot and why special precautions were not taken to avoid the need for such large corrections.

What is the justification for the authors' claim that the temperature is accurate to within ± 5° C. ? As shown by Figs. 13 and 14, the amount of heat lost by radiation and convection in 100 min. is small, therefore adequate cooling of the moulds between use is necessary if the bad effects of cumulative gains of heat are to be avoided. On this subject, operational investigations carried out at Appleby-Frodingham have indicated that ease of stripping is connected with the temperature of the mould.

Dr. **D. Binnie** (Lancashire Steel Corporation Ltd.) : When liquid steel fills the mould, the temperature of the inner surface of the mould will rise progressively. To know how much it rises is of importance in mould stress considerations and in determining the effect of temperature on ingot surfaces of both killed steel and rimming steel.

The authors should extend their work to ascertain the temperature of the inner mould surface at points met by the rising level of the steel.

Mr. **J. W. Grant** (British Cast Iron Research Association) : Mr. Heselwood and Mr. Pickering recorded growths of more than 6% caused by cyclic heat-treatment in their vacuum conditions. This is considerably greater than the approximate theoretical volume increase of 2% resulting from graphitizing of 1% combined carbon, which is the maximum likely to be present in the cast structure of an ingot mould iron. The 'vacuum' used by these authors, however, was only a condition of limited access of air and some oxidation took place. Results reported by M. Olette showed that in a more effective vacuum, growths caused by 80 cycles, 600–850° C., were nearly 5%.

At the British Cast Iron Research Association we have carried out similar tests in a vacuum with a pressure of 10^{-3} mm. of Hg—the same pressure as that used by Olette—and by cyclic treatment for 100 cycles between 600° and 900° C. we have obtained growths greater than 30% by volume. When samples previously annealed in air were subjected to the same cyclic treatment, the growth was only 2%. Evidently, the penetration of air into the test piece during the isothermal anneal had prevented, at least temporarily, the growth which occurs with the as-cast material.

Monsieur **J. Durand** (Compagnie des Forges et Aciéries de la Marine et d'Homécourt) : Dr. Binnie has referred to the desirability of knowing the temperature of the inner wall of the ingot mould.

On the advice of Mr. Malcor, we screwed an 18-mm. annealed high-carbon steel with coalesced cementite, through the wall of the mould and examined the change in the structure of the annealed metal. By this method we found that the temperature of the inner wall of a big-end-up ingot of 1400 kg. was slightly above the Ac_1 point.

AUTHORS' REPLIES

Mr. **J. Woolman** (Chairman of the Stresses in Moulds Panel) replying to the discussion on behalf of the authors of the papers in the Symposium on Stresses in Moulds said : It is unfortunate that sufficient progress has not yet been made to apply our results to ingot mould design, but a method of assessing the external surface stresses in a mould has been achieved. By applying this method to moulds of different designs it should be possible to evaluate the effects of any change and eventually obtain data to enable moulds to be designed scientifically.

Mr. Bacon gave some interesting figures regarding the degree of correlation between the life of moulds and the calculated growth based on the formula of Heselwood and Pickering. We should like to know how these moulds failed. A good correlation between the life of moulds which failed by crazing and the growth characteristics would be expected, but not between the lives of moulds which failed by major cracking and such growth characteristics, particularly if the moulds failed early in their life. In the case of moulds that failed late in life by major cracking, a certain amount of correlation might be expected as a result of the deterioration of the iron, which occurred on repeated reheatings.

Monsieur Olette states that the growth of the iron would tend to stretch the outside surface and therefore induce tensile stresses in the outer surface of the mould. We showed in our work that any such stresses could only be of negligible magnitude, because of the very rapid rate of relaxation of stress occurring at temperatures at which growth took place.

Mr. Gray referred to ingot cooling being accelerated by thicker-walled moulds. Lightfoot,* however, has shown that the rate of cooling of an ingot in a 4-in. thick mould was almost identical with that in a semi-infinite mould. The effect of greater wall thicknesses on the rate of solidification of the ingot was, therefore, very small.

Since the paper on strain measurements was written, further tests have been carried out on round moulds. The moulds were of the small-end-up type, 72 in. long, 16¾ in. dia., at mid-height. Two moulds had a wall thickness of 6 in. ; two were similar moulds but with 1½ in. machined from the outside of the wall, reducing the wall thickness to 4½ in. and the mould-weight/ingot-weight ratio from 2·0 to 1·4. The tests were on the moulds at their first usage, as before. The ingots were bottom poured. In the first test, one mould of each wall thickness was filled to a height of 54 in. only, the gauge sites being 46 in. from the base. In the second test, the height of filling was 43 in., with the sites 33 in. from the base. These are rather abnormal conditions.

The testing technique described in the paper was used, except that temperatures were measured at one point only on each site, using a potentiometer ; the results also were calculated in the same way. With the 6-in. thick walls the largest stresses found in the two tests were 53% and 54% of the breaking stress respectively, occurring after about 8 min. from start of teeming. With the 4½-in. walls the largest stresses were 60% and 59% respectively. In all cases these stresses fell only slightly during the 30 minutes of the test, by which time the surface temperature of the moulds had reached about 300° and 420° C. respectively for the 6-in. and 4½-in. walls. In contrast to the earlier tests the major stresses were approximately in the vertical direction throughout the test, although the differences between the major and minor stresses were again small. These results confirm the theoretical work of Land : lower stresses were found in the thicker-walled moulds. In both cases the stresses were lower than those found in the moulds described in the paper and it therefore seems unlikely that the reduction of the wall thickness from 6 in. to 4½ in. will lead to failure of these moulds by cracking. The moulds have not yet completed their lives.

Mr. **W. C. Heselwood** and Mr. **F. B. Pickering** wrote in reply : It is gratifying that Mr. Bacon has found, when applied to a large number of ingot moulds, a correlation between life and susceptibility to growth as calculated from the formula derived in our paper. Mr. Bacon's curve *a* (Fig. *B*) relates to moulds made in the same foundry as the irons from which the formula was obtained. We have more recently carried out growth susceptibility tests on irons from foundry *B* and found that a similar but not identical relationship existed, a factor of 0·75 having to be used with the formula derived for foundry *A*. The fact that the general levels of growth and of mould life are both less than for foundry *A* confirms Mr. Bacon's conclusion that there is some other important factor differing in the two foundries.

Just how susceptibility to growth may operate in lowering mould life is still the subject of discussion. Mr. Woolman has rightly pointed out that at the temperatures at which growth takes place, relaxation would occur almost immediately and so would not leave the mould in a state of stress that might be responsible for major cracking. Relaxation, however, implies distortion and it may be supposed that any form of distortion is likely to be detrimental to mould life ; as mentioned by Monsieur Olette, it may be related to the distortion and dislocation originally described by Benedicks and Löfquist.

It has been particularly interesting to learn that Monsieur Rocquet and Monsieur Olette have simultaneously been working on the same subject as ourselves and that their results are in general agreement with our own. In their cyclic tests they found considerably increased growth when the top temperature of the cycle was above the Ac_1 temperature and this is in conformity with our own results, which showed that, although under our conditions, little or no growth occurred at such temperatures, the fact that the iron had reached such temperatures could profoundly increase the degree of growth occurring later at lower temperatures.

The contributions of both Monsieur Olette and Mr. Grant on the effect of oxidizing atmosphere confirm earlier views that growth is not a simple problem but can probably take place by three distinct mechanisms : (i) graphitization ; (ii) disruption (as described by Benedicks and Löfquist) ; and (iii) oxidization. Factor (i) alone cannot account for growths greater than about 2% by volume, but can be accompanied by factors (ii) and/or (iii), both of which can account for large degrees of growth. Under good vacuum conditions, (ii) can proceed to an extraordinary degree, as shown by the 30% growth reported by Grant. Under conditions of limited access to air this growth is reduced, probably by a cushioning effect of oxide films around the graphite. In highly oxidizing conditions, however, progressive oxidation can again produce large growths, and the correlation reported by Olette between silica content and growth is significant.

Dr. **R. T. Fowler** said in reply : Mr. Gray referred to the use of surface pyrometers in this type of work. We at B.I.S.R.A. are attempting to use surface pyrometers

*N. M. H. Lightfoot, *Iron Steel Inst. Spec. Rep. No.* 2, 1932, p. 162.

more and more, for in spite of the advantages of the thermocouple technique it is a clumsy method, and it is applicable to only one mould at a time unless you have, say, ten calorimeters in the pit ; this is verging on the preposterous. We intended to measure the heat lost from the ingot at various times from teeming to stripping by moving the test mould into various positions in the pit, and in this way obtain a statistical relation between the influence of pit position and the influence of the temperature rise on the mould surface, and between the effect of initial steel temperature and the effect of initial mould temperature. If such a relation exists between the surface temperature rise on the outside of the mould and the heat lost from the steel, then one thermocouple can be placed at a convenient position on the mould surface, which is used as a reference point.

The next step would be to dispense with the surface thermocouple and use a contact pyrometer, so that in practice the surface pyrometer could be placed on a selected point on the mould surface, the temperature rise measured, and the heat loss from the ingot estimated from certain graphs. Knowing this heat loss, it would then be possible to estimate how much heat has to be put back into the ingot to raise it to rolling temperature.

In the last year we have done a number of runs using this test mould and have obtained some very interesting information. In this more recent work we went right through the mould wall to within $\frac{3}{64}$ in. of the inner face. We found that the temperature rose rapidly to about 700° C. in 10 min., and after 100 min. it had levelled out at about 850–900° C. on the inner face.

In our recent work* we have attempted to estimate the temperature distribution through the ingot. Starting with the tapping temperature, there is a slight fall in temperature in the ladle, and then a fall of surface temperature, at first steep and then more gradual, until after about 120 min. from the start of the tap, the surface of the ingot is at an average temperature of about 1050° C. If time-from-tap is plotted against heat content there is a slight fall in the ladle and then a rapid fall. We found that the ingot reached what we took as a representative value for the rolling temperature, 1300° C. (about 8 therms/ton, corresponding to about 208 cal./g.) in about 130 min., so that the heat content of the ingot was down to rolling heat in 130 min. from the start of the tap.

To simplify our calculations we assumed that the steel centre remained at the solidus temperature until it solidified, when there was a rapid fall in the temperature at the centre. For ingot heat conservation, it is necessary to attempt to strip the ingot before the centre has completely solidified, because, if it is left until after the centre has solidified, the removal of 0·5 therm/ton, which may only mean about 10 min. in the mould, will quickly bring the centre below 1300° C. If the centre is below 1300° C. and has to be brought back to 1300° C., a certain amount of heat has to be conducted through the entire mass of the ingot. It is better, therefore, to strip the ingot just before it goes solid, when there is a reserve of heat energy in the centre of the ingot, so that when placed in the soaking pit the centre cools to rolling temperature and the outside warms up to rolling temperature, rather than to bring both outside and centre up to rolling temperature, with all the attendant heat requirements to conduct the heat right through the mass of the metal.

Regarding Mr. Brooke Freeman's remarks about mild-steel plugs and cast-iron plugs, we changed from the former to the latter to determine whether there was any effect resulting from the different thermal properties of mild steel and cast iron.

We do not consider that the flattening of the curves, referred to by Mr. Turner, has any significance, because we can produce a set of four curves which do not show it. The terminal boards used were attached to both sides of the mould and were the ones used previously by Brancker,† who did not have automatic recording but used millivoltmeters. There may have been an error resulting from the difficulty of applying corrections for variations in resistance. We therefore changed to a recording potentiometer and our compensating leads were attached to the terminal boards on the side of the mould. We agree that these boards were a source of error, but in subsequent work they were removed and the cold junction of the system was located on the side of the pit wall, away from interference from heat, etc. In later work we found that on the pit side the temperature of the cold junction never rose much above 30° C. for most of our runs, but in the work where the terminal board was on the side of the mould the temperature of the board rose to about 300° C., as we show in Fig. 4, p. 285. We measured this temperature by having two thermocouples on the terminal board, one in the centre and one at the end. We estimated that the temperature distribution down the board was approximately linear, and a series of corrections could be applied to all the thermocouples which were connected along the side of the board. In our recent work, when we took the cold junction of the system to the pit wall and checked the new set of data with the set shown on p. 285, we found that the curves could be within the accuracy stated.

Mr. **A. Jackson** said in reply : The redesign of the moulds is based on information and experiences discussed at The United Steel Companies' Ingot Moulds Sub-Committee, on which Mr. Bacon serves. Our present stripping time of 50 min., and mould life of 165, fall exactly on the curve given by Mr. Bacon (Fig. *A*).

The mould temperatures were obtained to enable an assessment to be made of the time required to cool the mould to about 50° C. The simple method used renders a comparison with the multiplicity of figures obtained by Fowler and Stringer extremely difficult.

The narrow side of the mould is $\frac{1}{2}$ in. thinner than the broad side—this accounts for more rapid heating. The figures show that, later, the broad-side temperature exceeds the narrow-side, because of increasing radiation from adjacent moulds.

It is difficult to assess the fuel saving at the soakers resulting from more rapid stripping, since many months are required to build up production. Throughput, from four old soakers, has increased, however, by over 1000 tons of ingots per week, on a previous average production of 10,300 tons. Present gas consumption is 9·5 therms/ton and this includes the charging of 900 tons of cold ingots per week. The hot ingots are charged into unfired Gjers pits until required for rolling ; these pits reduce heat losses to about half the losses in the open air.

When a mould is nearing the end of its life, its inner surface becomes crazed, thus preventing very rapid stripping (less than 50 min.). This effect is more marked when the mould is re-used at increasing temperatures. This difficulty is being overcome by lightly dressing the inner surface of the mould with a grinding wheel after it has passed half life.

About 20 years ago Mr. Turner produced some information on the rate of cooling of ingots. Since much of this information is not generally known even today it should be recovered from his archives.

*Paper PE/A/39/51 : British Iron and Steel Research Association.

†*J. Iron Steel Inst.*, 1950, vol. 165, pp. 307–313.

Joint Discussion on the Papers—

METALLOGRAPHY OF CARBON IN SILICON–IRON ALLOYS CONTAINING 4% SILICON

*By E. D. Harry**

THE VARIATION IN ELECTRICAL PROPERTIES OF SILICON–IRON TRANSFORMER SHEET

By S. Rushton and D. R. G. Davies†

Mr. **E. D. Harry** (Steel Company of Wales Ltd., Lysaght Division) presented the first paper, and Mr. **D. R. G. Davies** (Richard Thomas and Baldwins, Ltd.) the second.

Dr. **R. V. Riley** (Staveley Iron and Chemical Co. Ltd.) : Mr. Harry, on page 245, comments on the apparently large amount of carbide phase in the microstructure of a 4% silicon 0·01% carbon steel, which "seemed to be rather more than would be expected if a plain carbon steel containing a similar amount of carbon is taken as a basis of comparison." This agrees entirely with my own observations on iron–carbon–silicon alloys of much higher silicon content and higher carbon content, and it may indicate that the carbide does not contain a sufficient percentage of carbon to give cementite, Fe_3C. I have found that the average carbon content of the silico-carbide phase is between 2% and 3·5%, not the 6·6% required if the phase had the composition of cementite.

The author states that the two carbide phases are completely insoluble, or show only limited mutual solubility at room temperature. Whilst this may be true, it does not indicate that the silico-carbide phase has a fixed silicon content, at least not in the higher silicon range of iron–silicon–carbon alloys. Hurst and Riley‡ have shown that this phase exists in commercial 10–15% silicon irons, and that the silicon content of the carbide phase is related to the silicon content of the whole alloy. The silico-carbide phase may dissolve silicon in excess of the normal composition of the phase. Piwowarsky§ found that with iron–silicon–carbon alloys containing between 4% and 16% silicon a magnetic transformation as well as the A_2 transformation, occurred at a temperature in the range 580–450° C., that depended on the silicon content. This supplementary transformation was attributed to an unknown compound formed from iron silicide, Fe_3Si, and an excess of either iron or carbon. The magnetic transformation of cementite was observed in normally cooled alloys only up to 5% silicon. If the double compound of carbide and silicide was heated to between 700° C. and 1000° C., graphite was precipitated and the magnetic anomaly disappeared ; the stability was less than that found with cementite in alloys of low silicon content, but became increasingly stable with increasing silicon, especially over 10%. This, I think, confirms the presence of a double carbide as described by Mr. Harry.

In the paper by Rushton and Davies I was interested in the heterogeniety reported in the ingot. Steelmakers have considerable difficulty in casting ingots, and I wondered why they wanted to make one. They will make transformer sheet, a rolled sheet product, out of it, so why not make it in sheet form to start with ? A possible way of removing the non-metallics is to cast the steel centrifugally into moderately large cylinders, which are then split longitudinally, opened out under a press, and squeezed down into a plate. The side which has been on the inside of the centrifugally cast cylinder will contain the undesirable non-metallics which can be removed by machining. The sheet can then be reheated and again rolled to sheet form. This is a development of the method of blowing ordinary window glass ; the glass-blower blows a cylinder, opens it out, and then presses it flat.

The author gives the percentage carbon content of sheet samples to the third decimal place. Is the barium hydroxide method of analysis sufficiently accurate to justify this ?

The **President** (Mr. R. Mather) : Dr. Riley's suggestion of a new method of casting the steel should give many of us, including the Engineers Group, something to think about.

Mr. **A. D. Grace** (Guest Keen Nettlefold Research Laboratories) : A statistical approach does not in any way invalidate the conclusions drawn by Rushton and Davies but the following points may be of interest.

During the examination of watts loss, was any allowance made for the variation of gauge between individual sheets ? I have found that with the 0·014-in. hot-rolled sheet, this variation can cause a difference in total loss of about 0·02 W./lb. as a result of the change in the eddy current component. This may, to some extent, account for part of the observed variation between sheets of a pack, but not for all.

In the case of the two laboratories making estimates of carbon content, not only are the laboratories apparently giving significantly different results, but the samples themselves are different. The numbers are rather small, however, for forming a definite conclusion.

The authors refer to the two significant factors which contribute equally to the variation. I rather disagree with the word 'equally' because, although the two mean-squares in the analysis of variance are of the same order of magnitude, the components of variance show that ingot variation accounts for about 38·5% of the total and position in pack for about 12·6%, leaving an unexplained residual of 48·9%.

Mr. **H. H. Stanley** (The Steel Company of Wales Ltd., Lysaght Division) : Rushton and Davies have shown that, with the process which they describe, very wide variations in the magnetic properties of the product may occur, which suggests that the manufacture of transformer sheets is difficult to control. The work which they describe has involved the destruction of a large number of expensive sheets, and it is unfortunate, therefore, that a greater number of metallurgical tests

* *J. Iron Steel Inst.*, 1951, vol. 167, March, pp. 241–246.

† *Ibid.*, pp. 247–261.

‡ J. E. Hurst and R. V. Riley, *Ibid.*, 1944, No. I, pp. 213P–218P.

§ E. Piwowarsky, *Stahl u. Eisen*, 1925, vol. 45, p. 634.

was not carried out and reported together with the statistical study.

Unfortunately only two sheets were analysed, but I am very surprised to find that the authors give the carbon analysis of the cast as 0·05%, and of the two sheets as 0·040 and 0·045%. It is very difficult to keep carbon in thin sheets during hot rolling in packs, and it is very unusual for there to be virtually no removal of carbon during pack rolling. I would not expect to find more than 0·02% of carbon in the sheets after hot rolling. The authors have suggested that decarburization explains some of the variation they find in the core losses. It is very surprising, therefore, that the carbon contents should be almost the same in two samples showing such different values of core loss as 0·94 and 1·40 W./kg., and I wonder whether the analysis is completely reliable. I have never found core losses as low as 0·94 W./kg. with carbon contents higher than 0·015 to 0·020%.

During micro-examination of the samples, did the authors note the presence of graphite? Mr. Harry, in his paper, states that with low-temperature annealing (750–800° C.) if the carbon is in excess of about 0·012% most of it is in the form of graphite. The authors claim that microscopical examination showed no significant difference in structure between any of the samples with varying core losses; the variations within samples were so great that they masked any variations between samples. This suggests that very wide variations exist within one sheet. My own experience with this type of material is that the carbon and grain size, in particular, would account for most of the serious variations found in sheets rolled from the same cast.

Over a number of years, I have carried out many investigations into the sometimes appreciable variation in magnetic properties resulting from the position in the pack. The variation is apparently a characteristic of the mill. Each unit will, in general, give a characteristic relationship between core loss and position in the pack. The authors show that after the second anneal the relationship may be represented by a **U**-shaped curve. I have found a similar relationship, and also one that could be represented approximately by a straight line which sloped gradually from No. 1 sheet up to No. 8. I have also found, when mill conditions were very good, a horizontal straight line, which indicated very little variation resulting from difference of position in the pack. In Fig. 7, the range of average core loss is represented by the extremes of No. 3 sheet and No. 5 sheet, and half the range is given between No. 4 and No. 5 sheet. Why should there be such a great difference between No. 4 sheet and No. 5 sheet? If the process is considered at an earlier stage, Nos. 4 and 5 sheets are seen to originate from the same sheet by the operation of doubling, No. 4 sheet having been turned upside down. Thus, turning a sheet upside down would appear to give a marked improvement in the core loss; I suggest, however, that it is caused by the variation already existing between the two ends of the sheet before doubling. The authors apparently did not measure the variation in core loss between one end of a sheet and the other, because they used the whole sheet in their core loss test.

It is suggested that the differences between the ingots are probably caused by non-metallics, some of which are so small that they cannot be seen under the microscope. The authors also referred to the wearing away of the stopper nozzle, and suggest that this might be one of the causes of the variation from ingot to ingot. Non-metallics, which originated from the stopper nozzle, would surely be visible under the microscope, and more careful micro-examination would have shown relationships caused by non-metallic inclusions.

The data which the authors have given show wide variation in core loss over one cast. Is this representative, or is it abnormally large? It is certainly much larger than I have found over a number of casts. Some indication of the origin of the sheets that gave extreme results would have been very interesting.

Dr. **M. L. Becker** (British Iron and Steel Research Association): Mr. Harry has given us some very beautiful but apparently rather mysterious microstructures. In his interpretation of these structures I think that he tends to overlook the possibility that his alloys were heterogeneous before cooling from the annealing temperature. Commercial silicon sheets, when examined at room temperature, may often look as homogeneous as pure iron–silicon alloys, but in fact they are not. A very small amount of carbon added to an iron–silicon alloy will considerably alter the shape of the gamma region of the iron–silicon equilibrium diagram, as shown by Yensen and by Rickett and Fick, whose findings are reproduced in Figs. 24 and 23 of the paper. In commercial silicon sheet, nitrogen and manganese are present as well as carbon, and the effects of these would be expected to be additive.

I believe, therefore, that in commercial transformer sheet there are usually two massive phases of different composition present at the annealing temperature. One of these will be a ferrite phase containing a high percentage of silicon and a low percentage of carbon, the other will be an austenite phase, with higher carbon and probably lower silicon contents. The heterogeneous condition established at the high temperature would not be expected to disappear during even quite slow cooling and the resulting microstructures are to be interpreted accordingly. For example, the grain-boundary carbide shown in Figs. 7 and 8 probably resulted from the presence of grain-boundary austenite during the anneal. Similarly, the large area of pearlite shown in Fig. 9 was probably an austenitic region at the high annealing temperature. There are many other examples.

Mr. **H. Morrogh** (British Cast Iron Research Association): Mr. Harry states that when the carbon content of the low-temperature annealed sheet is more than 0·012%, graphite is invariably present, and that when it is less than 0·006% no free carbide can be detected. Can this not be explained by the presence at this temperature of ferrite saturated with carbon, and in some cases free, relatively unstable, carbide? In the presence of so much silicon, carbide would decompose fairly readily, so that, in all samples containing more than a certain percentage of carbon, graphite would be found, and in samples containing less carbon no graphite or iron carbide would be visible.

Did the author experience any grain growth during this low-temperature annealing and, if he obtained such grain growth, was it affected by the presence of graphite? In the production of malleable cast iron, we find that grain growth of ferrite is almost completely inhibited if graphite is present, but if that graphite is removed, for instance by decarburization, ferrite grain growth occurs automatically.

When describing the migration of carbon to the surface of the sheet, the author suggests that the silicon near the surface of the sheet may have been oxidized, giving a thin film low in silicon. He has not used a spectrograph to confirm this but assumes that the material is likely to be completely ferritic at the temperature of annealing. If pearlite is present austenite must have been present at some time during the heat-treatment. If the material is in fact desiliconized, austenite could be formed by enlarging the γ field. The isothermal diagram of this system shows that the concentration of carbon in the austenite, under equili-

brium conditions, must be higher than that in the ferrite, so that one would expect the carbon content of the austenite during the annealing to be, as a result, higher than that in the ferrite, and, where it has been austenitic, pearlite to develop.

The authors' tentative conclusions (5) and (6), that the two carbides appeared to be completely soluble in each other at high temperatures, and to undergo a eutectoid reaction at a temperature of about 400° C., have not been confirmed. Another cut through the area of the micrograph given in Fig. 21*b* could show a particle which contained none of the duplex structure, the particle with the duplex structure shown in Fig. 21*a* being above or below the surface of the new cut. Even a careful study of the paper did not reveal any evidence that would substantiate the existence of a eutectoid reaction or its occurrence at 400° C.

The silico-carbide found by the authors in these alloys containing 4% silicon confirms my observations on the paper by Owen, that it is incorrect to assume that the carbide in equilibrium in this system is Fe_3C.

Mr. **R. Rawlings** (English Electric Co. Ltd., Nelson Research Laboratories): I agree with the last speaker that where there is pearlite on the surface of the sheet, as in Fig. 10, austenite must have been present on the surface. As the author suggests, during vacuum annealing silicon is oxidized, and probably vapourized as well, at the surface, and when the silicon content is sufficiently lowered austenite is formed. We have found a definite decrease in silicon, and have detected silicon in other parts of the furnace. Carbon will diffuse from the ferrite into the austenite until a temporary equilibrium is established. This will not be a true equilibrium, because of the silicon concentration gradient. The final ratio of carbon concentration in the austenite to that in the ferrite may be expected to be in proportion to the solubilities in the two phases. Further oxidation of carbon at the surface will be prevented by the formation of a continuous silica film.

If annealing were continued at 1100° C., in dry hydrogen, to prevent further oxidation of silicon, the silicon concentration gradient would level out. The high carbon concentration near the surface, however, would maintain the austenite phase and hence its own continued concentration near the surface. We should thus have an alloy being annealed at 1100° C., not in equilibrium and apparently not tending to equilibrium. Apart from removal of the carbon at the surface, equilibrium can apparently only be achieved by annealing below the A_3 or above the A_4 transformation point of the high-carbon material. Similarly, the other examples quoted in the paper may be explained by the formation of austenite in the low-silicon areas.

The segregation of carbon to the low-phosphorus bands in mild steel, can also be similarly explained. During cooling, the areas high in phosphorus transform first. At some temperatures the high-phosphorus areas will be ferritic while the low-phosphorus areas are still austenitic, causing carbon to diffuse from the ferrite to the austenite. At lower temperatures, after complete transformation has taken place, diffusion of carbon is too slow to distribute the concentrated carbon and thus bands of pearlite remain.

It is a pity that an attempt was not made to extract the silico-carbide and analyse the residue, as its presence supports the view of Marles.* From what evidence does the author conclude that the eutectoid change takes place at 400° C. ?

The author states that the equilibrium diagrams based on pure alloys may not apply to commencial steels. Can he therefore give a fuller analysis of the steels used, including nickel, copper, chromium, and nitrogen ? Nitrogen may have just as great an effect as carbon. What effect would he expect the 0·1% copper and 0·1% nickel, usually found in silicon iron, to have on the existence range of the γ loop ?

The use of centrifugal casting mentioned by Dr. Riley has already been seriously considered, and discarded. The effect of inclusions on magnetic properties varies with the size of the inclusions. Dijkstra and Wert† have recently shown the existence of a certain low optimum size, by precipitating iron carbide at low temperatures in different-sized spheres. This size, 1200 Å., would probably not be removed by centrifugal casting.

I think it would have been of slightly more value if Rushton and Davies had used hysteresis loss, and not the total watts loss, as an indication of quality. The total loss includes the eddy-current loss and hence any variations caused by differing silicon content and sheet thickness. Hysteresis loss is not affected by such variations.

Mr. **H. Brooke Freeman** (John Summers and Sons Ltd.): I suggest that Mr. Davies has not given the rolling details sufficiently fully to be able to check variations which can be caused by slight modifications of heating and rolling practice. When rolling silicon sheets, the watts-loss figures are modified by such practice as rolling a smaller number of packs in any one heat, by the temperature at which these packs are rolled, by the speed at which the sheets are changed, and the method of changing, by the method of reheating the packs in the running-over furnaces and by the number of passes the sheets receive in progressing from bar to final sheet.

The heating of the doubles and of the finished packs must be very carefully done. It is quite easy to get a cold end near the door of the furnace. When rolling 4% silicon sheets, it was found that it was good practice to get the bars hot and to maintain the moulds, doubles, etc., as hot as possible until they were in final sheet form. A good mill crew, working quickly to get doubles and eights back into the furnaces, can help considerably to reduce watts losses.

The **President** (Mr. R. Mather): I should like to thank all those who have presented papers for their contributions.

The last two papers have dealt with a subject that is of real importance in the economics of this country. I was concerned, three or four years ago, with the shortage of supply of high-silicon sheets. In those days there were considerable difficulties in obtaining collaboration between the relatively few companies that were producing them.

Mr. Stanley has drawn attention to the improvement in that position. I hope that these papers mark the beginning of some publicity on the technical problems of production. Today's discussion may have introduced new ideas to the representatives of the manufacturers of silicon sheets who have been here. I believe that improved quality can best be achieved by publication and discussion of the techniques involved in production. I thank the companies who have rendered such good service in this way.

A President of this Institute, Sir Robert Hadfield, proposed the use of high-silicon sheets for reducing these watts losses many years ago. The Institute should rightly take an active interest in the further develop-

* D. Marles, *J. Iron Steel Inst.*, 1948, vol. 158, pp. 433–436.

† L. J. Dijkstra and C. Wert, *The Physical Review*, 1950, vol. 79, No. 6.

ment of this subject, which is important in the general economy of this country, because it has a great influence on the rapidly growing production and use of electric power.

CORRESPONDENCE

Mr. **R. D. Hudson** (The Steel Company of Canada, Ltd.) wrote: What is the nominal analysis of the town's gas which produces the atmosphere surrounding the charge during the low-temperature annealing of transformer grade sheet? Mr. Harry states that "when the carbon content of this type of sheet is more than about 0·012%, graphite is invariably present and very little, if any, carbide is found." Is this phenomenon peculiar to the 750–800° C. temperature range? Is it confined only to this transformer sheet or would it also exist with 2·5% silicon sheet? Would there be any change if the rate of cooling of the charge were altered?

Most of the carbide is presumably in the form of grain-boundary films when there is a small grain size prior to annealing. Would this condition be satisfactory for the production of graphite in a low-temperature commercial anneal, or would the globular and acicular form of the carbide be more susceptible to graphitization?

What type of carbide would result if the commercial low-temperature annealing treatment was similar to the treatment of the laboratory sample annealed at 1050° C.? In the laboratory-annealed sample, the rapid rate of cooling prevented the complete eutectoid reaction. Would the electrical properties be different from those of a sample that had been slowly cooled? Would a duplex carbide be found in a 0·25% silicon sheet with 0·04% C after any method of cooling?

What does Mr. Harry consider is the distribution of carbon that is most conducive to maximum electrical properties for any grade of silicon sheet with up to 0·04% C.?

Mr. **Dunn** (Products Research, Liverpool) wrote: What method of deoxidation did Mr. Harry use in the manufacture of the iron? The addition of carbon to molten iron not only reduces the quantity of oxygen, but also renders the oxygen present less active. When the temperature is lowered, the solubility of oxygen in the metal is decreased, and equilibrium is re-established by the separation of an oxide phase, inclusions being formed when the oxide droplets are trapped by freezing of the metal before they rise to the surface. The addition of an element that can combine with oxygen in metal, *e.g.*, manganese, silicon, or aluminium, will increase the amount of oxygen that can remain in solution without the precipitation of an oxide phase. The inclusions that will eventually precipitate will be composed of that oxide which has exceeded its solubility in the metal, for example, the addition of manganese would produce (Mn.Fe)O inclusions. Inclusions, such as chromite and titanium cyanonitride, that have melting points above the temperature at which they precipitate from the steel* will usually form idiomorphic crystals that do not elongate when the steel is rolled or forged. Inclusions that are liquid at the temperature of precipitation, usually assume a droplet shape and often, although not always, deform during hot working of the steel. Many of the siliceous inclusions are of this nature. Some inclusions, such as silicate sulphides, are a single phase at the time of formation in the steel, but during cooling separate into two phases. Sometimes metal-inclusion eutectic structures are observed. Baker† on silicon–iron shows some photomicrographs of allotriomorphic crystals of ferrite, with a few patches of laminated pearlite and Zapffe‡ more recently shows the cluster-type silicate inclusion in deoxidized electrolytic iron.

Impurities such as carbon, sulphur, nitrogen, and oxygen occupy interstitial spaces in the crystal structure and even small amounts affect the magnetic properties of the metal and their low solubility at room temperature causes ageing. Yet pure iron–silicon alloys, produced by vacuum-melting techniques from hydrogen-fired electrolytic iron and 99·9% Si and having a total impurity level well under 0·1%, when processed and heat-treated by the standard methods used for transformer irons were found to be unsuitable because of uneven crystal growth. This appears to confirm the view of Feitknecht§ that some impurities are necessary.

Mr. Harry states that during high-temperature annealing the charge was surrounded by an atmosphere of hydrogen and nitrogen, which produced a large grain size, so that the whole thickness of the sheet was often covered by one grain. He does not state in what quantities the gases were used. Nitrogen has a retarding effect on the crystal growth of iron–silicon alloys, and at some critical content near 0·0015% it prevents their growth. Silicon contracts the gamma-iron field in the equilibrium diagram, so that the transformer irons possess no allotropic transformation, and can be annealed at high temperatures without recrystallization during cooling; thus large grains can be obtained. Silicon and aluminium can be added to iron in fairly large quantities without greatly affecting the magnetic properties, although more than 4·5% of Si or 8% of Al makes iron too brittle for most commercial uses. The main difficulty is that if the iron is not thoroughly deoxidized before the alloying elements are added, oxide inclusions are formed that cannot again be eliminated, and these have deleterious effects on the magnetic properties.

Mr. **Pugh** (Messrs. Joseph Sankey and Sons Ltd., Wolverhampton) wrote: I should like to comment on the apparent lack of decarburization during processing. Rushton and Davies give the cast analysis as 0·05% and yet the annealed sheet shows a reduction of only 0·01% or less on the cast carbon. In normal processing a reduction from 0·50% to 0·025% C from sheet bar to hard sheet would be expected, with a further reduction on annealing. I would have expected a total carbon figure in the final sheet to have been between 0·01% and 0·02%. An unexpected difference of performance between sample *A* and sample *B* is recorded; although they have high carbon percentages they give 0·94 W./kg. and 1·40 W./kg. It is unusual to get 0·94 W./kg. with a carbon as high as 0·044%. Were the samples upon which the carbon determinations were conducted truly representative?

I would have expected micro-examination to be very informative, and to show especially carbon content variations, and hence the effect produced on the grain size. A watts loss of 1·4 W./kg. rarely gives the same grain size as one with 0·94 W./kg. The difference in size is usually marked—often as much as 4 to 1 and higher. I think it unfortunate that so little evidence of the effect of position on the decarburization of the sheet was included in the form of carbon determinations. The results given imply that if a 0·94 W./kg. result can be obtained with what is almost the original cast-carbon percentage, then the importance of decarburization has been over-emphasized, and I am sure that this is not so.

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* Transformer iron is called 'steel' in the electrical industry.

† T. Baker, *J. Iron Steel Inst.*, 1903, No. II, p. 320.

‡ C. A. Zapffe, *Ibid*, 1946, No. II, p. 156P.

§ W. Feitknecht, *J. Inst. Metals*, 1926, vol. 1, p. 131.

The differences in results for different sheet positions in packs have been fully established, and one of the causes has been found to be a difference in sheet gauges, especially for the bottom, or No. 8, sheet, which generally rolls thicker than the rest. A statistical examination has shown that the lowest losses have come almost equally from the tops, middles, and bottoms of the various heats examined over a period of time. Annealing-pot positions would apparently not be significant except where all the sheets were made from the same cast and annealed together in the same pot.

In Mr. Harry's paper there are one or two points which apparently conflict slightly with my own experience. He states that the carbon in the sheet bar consists of pearlite and small amounts of carbide. We have found by chemical analysis that as much as 20% of the total carbon in the sheet bar is in the form of graphite. This applies to both open-hearth and electric arc-furnace steels. I have found (again by chemical analysis) that the percentage of graphite in the as-rolled or hard sheet remains substantially the same as found in the sheet bar.

Mr. Harry mentions graphite for the first time as being present in the low-temperature annealed sheet and states that it is there when there is more than about 0·012% C. He also found that this graphite is formed during annealing below Ac, only if the as-rolled or hard sheet contains pearlite. I have found that graphite has been present, and in almost constant quantity right from the sheet-bar stage and appears to be very difficult to remove. My observations are based on careful chemical analysis using 20-g. samples. Does Mr. Harry think that there might be a possibility, especially in examining specimens of the sheet bar and hard sheet, that this graphite might have been removed during preparation of the micro-specimens ?

AUTHORS' REPLIES

Mr. **Harry** wrote in reply : I would like to thank Dr. Riley for drawing my attention to the work of Piwowarsky.

I agree with Dr. Becker that nitrogen, manganese, and carbon extend the gamma loop in the iron–silicon diagram and considerable further work is required to evaluate the effects of these elements, which are always present in commercial iron–silicon alloys. The pearlite area mentioned by Dr. Becker must have been formed from austenite as there is no other way in which it can be produced. However, I do not think that the carbide films originated from austenite.

In answer to Mr. Morrogh's first point, it should be emphasized again that the statement that when the carbon content exceeds 0·012%, graphite is invariably present, refers only to sheets annealed at about 760° C. The figure of 0·012% is arbitrary and depends on the process employed for making the sheet. Samples annealed at 1150° C. contain as much as 0·035% of carbon in the form of carbide. Graphite is formed in the low-temperature (760° C.) annealed sheets only if the as-rolled sheet contained pearlite. I have not observed that the small amount of graphite present in these sheets has any pronounced effect on grain growth.

Mr. Morrogh and Mr. Rawlings discuss the diffusion of carbon to the surface. It was unfortunate that facilities for carrying out spectrographic tests were not available. If a skin, sufficiently low in silicon to be austenitic, was formed on the surface, then the mechanism described by these speakers would, of course, operate. However, even if the silicon content of the skin was not sufficiently low to produce austenite by its own effect alone, a slight diffusion of carbon from the higher silicon ferrite in the body of the sheet would soon produce austenite in the skin. Since there is pearlite present, austenite must have been formed by some means. If desiliconization alone is capable of producing an austenitic skin on the surface, then evidence of it should be found in a sample which was originally free from carbon. I have never observed anything that resembled a skin of smaller grains on the surface of samples, except where intergranular oxidation has taken place.

I agree with Mr. Morrogh and Mr. Rawlings that further proof of the eutectoid reaction is required. Mr. Rawlings' remark regarding volatilization of silicon is entirely confirmed, and his description of a system remaining in a state of non-equilibrium during annealing is very ingenious. The elements which Mr. Rawlings mentions are present in the sheets in the following amounts—Ni 0·05%, Cu 0·08%, Cr 0·02%, N_2 0·003%.

These remarks will also answer Mr. Hudson's question regarding the presence of graphite. I have never found graphite in 2·5% silicon sheets. I cannot suggest what the effect of the two carbide phases might be on the magnetic properties. I would not expect to find the duplex carbide in a 0·25% silicon alloy.

It would be very difficult to define the least harmful form of carbon, but from considerations of the distribution of a second phase, graphite would appear to be least harmful.

Mr. Dunn's remarks are very interesting, but extend rather outside the subject of the paper.

Mr. Pugh's statement that he has found graphite in sheet bar and as-rolled sheet is exceedingly interesting. I have not observed, metallographically, any graphite in either the sheet bar or as-rolled sheet. I wonder if Mr. Pugh has examined his graphitic residue by X-ray diffraction ?

Mr. **S. Rushton** and Mr. **D. R. G. Davies** wrote in reply : We would like to thank Mr. Stanley, in particular, for his observations. The cast examined was chosen for special study at the beginning of melting and not after the test on the basis of the results being most representative of the normal product. The cast is, in fact, slightly poorer than normal for the period during 1947, when it was made, and is very much below the average quality being produced today with modified procedures. An important point is the number of samples taken. When so many tests are taken a wide range of iron loss values is to be expected, and if Mr. Stanley were to test on this extensive scale he too would probably find variations far wider than his usual procedure leads him to regard as normal.

The tentative suggestion made in the paper that differences between ingots might be partly caused by refractory material falling from the nozzle into the mould is reasonable but, as Mr. Stanley states, is not supported by any microscopic evidence. He would, no doubt, agree that material worn from ladle nozzles must enter the moulds, and the effect of such non-metallics could not be other than harmful to electrical properties. It is conceded that, taking the investigation as a whole, more extensive metallographic work might have been informative, particularly with respect to surface conditions of the finished sheets. This was well appreciated after the iron loss results had been examined, and was mentioned in the conclusions to the paper.

Both Mr. Pugh and Mr. Stanley have expressed surprise at the results of carbon analyses quoted ; at the time when these figures were obtained there was a good deal of disagreement among chemists as to the most reliable method of determining such small amounts of carbon. All are agreed that, in general, low carbon is

advantageous. There is also agreement that large grains are in general more favourable than small ones but Mr. Stanley's claim, that carbon and grain size alone account for most variation within a cast, is very unexpected. It would have been more convincing if he had produced figures in support of this.

Mr. Stanley's confirmation of the fact that for a particular mill furnace there is a characteristic pattern in iron loss values when related to the sheet position in the rolled pack is most interesting, and it is especially helpful to know that in one study, when conditions of good control were achieved, it was possible to produce a horizontal straight line characteristic—or, in other words, a situation where variations between the sheets in the pack were substantially eliminated.

In the paper no explanation was brought forward for the differences between positions 4 and 5, and this might have been achieved, as Mr. Stanley has suggested, if surveys along each sheet had been carried out. This was impossible because at the time no single strip tester was available, and each Churcher pack consumed almost the whole of a sheet.

Mr. Grace has pointed to the effect of variations in gauge, and Mr. Pugh is also to be thanked for giving evidence on this point. There is no doubt that sheet thickness has a bearing on iron loss values although the relationship is not simple, particularly in the range 0·012–0·015 in., and it is by no means easy in a works experiment to isolate the effect of gauge from surface effects which, from the very nature of the sheet rolling process, are likely to be correlated with thickness. Mr. Rawlins would have avoided the gauge difficulty by using hysteresis loss as a criterion of quality, but in our works study this would not have been convenient nor in some ways so informative because, in the trade, quality is assessed on total iron loss. We cannot appreciate that the choice of hysteresis loss would have eliminated the influence of variations in silicon.

Mr. Grace is quite correct in his remarks concerning the contributions to total variation in ingot 2 of the two significant factors: ingot position and sheet rolling position. As far as the figures for carbon analysis are concerned, they do indicate significant differences between samples and between laboratories, as Mr. Grace suspects.

Mr. Freeman has raised a number of points relating to the adequate heating of packs during sheet rolling. They are points well appreciated by experienced rollers and the fact they were given adequate attention was confirmed by a team of observers during the experiment. Their observations are covered in the section of the paper dealing with sheet rolling.

Joint Discussion on the Papers—

THE CRYSTAL STRUCTURE OF GRAPHITE IN CAST IRON

*By W. S. Owen and B. G. Street**

THE CARBIDE PHASE IN IRON–CARBON–SILICON ALLOYS

By W. S. Owen†

Dr. **B. G. Street** (Department of Metallurgy, University of Liverpool), in presenting both papers, added: The work reported in these papers was carried out nearly four years ago. During this time, the X-ray diffraction techniques used have been found to be slightly less accurate than was previously believed. For example, it was not possible to distinguish between the cementite parameters given by Hume-Rothery and those given by Lipson. Although we believe that the conclusions drawn are still substantially correct, if the work was repeated today a refinement of the diffraction technique, such as the use of a crystal monochromated beam, would ensure higher accuracy in the results. The work was carried out as a step towards the experimental determination of the iron–carbon–silicon diagram. Later results, which have been submitted for publication, fit in with and substantiate the earlier conclusions.

Since the work reported in the second paper, we have examined several iron–carbon–silicon alloys containing cementite; in every case the diffraction pattern has been indistinguishable from that of Fe_3C. We feel that this is good evidence in support of the argument that the amount of silicon entering the cementite must be negligibly small. The validity of the subsequent arguments concerning the relative arrangements of the phase fields in the ternary equilibrium diagram depends upon the sensitivity with which the method would detect changes in the composition of the cementite. Although we believe that the data is sufficiently accurate to allow a re-examination of the equilibrium diagram, it is nevertheless necessary to obtain confirmatory evidence.

Professor Hultgren and Mr. Kuo have contributed results obtained by chemical analysis of extracted carbides produced by tempering a hardened silicon steel (*see* p. 240); these results show that in the later stages of tempering silicon is not present in the cementite. They also mention the magnetic work of Tammann and Ewig.[1] Recently, at the Massachusetts Institute of Technology, we have carried out magnetic measurements on hardened and tempered high-purity silicon steels, and have obtained results similar to Professor Hultgren's. For example, using the ballistic magnetometer described by Zmeskal and Cohen,[2] a steel containing approximately 1% of C and 2% of Si has been examined after hardening by quenching in water followed by refrigeration in liquid nitrogen and tempering by continuous heating at a rate of approximately 5° F. per minute up to 700° C. After this treatment the steel consists of fine graphite flakes in a ferrite–cementite matrix. It is thought that this method of producing cementite would be most likely to cause silicon to enter the structure, should this be possible. The Curie point on slow cooling was found to occur at 209° C., which is the same as that found for a fully annealed plain carbon steel and is in good agreement with the figure given by Crangle and Sucksmith[3] for the Curie point of pure Fe_3C. A similar experiment on a steel with a rather high carbon content (so that some cementite did not dissolve during the austenizing treatment) gave the same result to within 1° F. The Curie temperature is usually considered to be very sensitive to small changes in composition; these results, therefore, appear to support the conclusions drawn from the X-ray diffraction work.

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* *J. Iron Steel Inst.*, 1951, vol. 167, pp. 113–116.
† *Ibid.*, pp. 117–120.

No evidence has been found to suggest that silicon enters the cementite structure, but we have produced evidence to show that in a high-silicon (8%) low-carbon (1%) alloy, a carbide constituent containing silicon can form on slow cooling. This carbide has a crystal structure very different from that of cementite. Annealing causes these alloys to graphitize; the carbide therefore cannot be considered an equilibrium phase and so does not need to be taken into account when discussing the general form of the equilibrium diagram.

The usual form of the ternary diagram is shown as a basal projection in Fig. 4 of the paper; as illustrated in Fig. 5, if the point *C* is placed at the composition Fe_3C (which the experimental work has indicated to be the correct position), the four-phase plane will contain a re-entrant angle. Recently, we have examined the relevant part of the system by a microscopic study of quenched specimens and by thermal analysis of high-purity alloys cooled at a slow controlled rate in a vacuum furnace. The results have been incorporated in a paper by Dr. Owen and J. E. Hilliard which is to be published at a later date. They find that the *L* point lies on the high-silicon side of the α point, and not on the low-silicon side of the γ point as deduced by previous investigators. This result allows the *C* point to be placed at the composition of Fe_3C and the four-phase quadrilateral to be constructed without including a re-entrant angle.

Dr. **R. V. Riley** (The Staveley Iron and Chemical Co., Ltd.): Although appreciating the high degree of accuracy with which the work has been carried out, and applauding the methods used, I feel that some of the conclusions stated by the authors are rather sweeping.

In the paper by Owen and Street, the conclusion (p. 116) that "The crystal structure of any particular microscopic form of graphite is independent of the composition and method of manufacture of the iron," appears hasty, in view of the limited number of specimens of cast iron examined. The specimens of cast iron mentioned in Table I presumably include none containing more than 3–4% of alloying elements such as Ni, Cr, Mo, or even Si. The statement (p. 114) that "A wide variety of cast irons, containing graphite in many different metallographic forms, has been investigated," is therefore also misleading.

Conclusion (4) infers that the pure graphite, the material extracted from the cast iron, does not contain any element other than carbon. After the very extensive purification treatment accorded to the extracted graphite, this is to be expected. Presumably this conclusion relates only to the *pure* material, and not to the graphite as it exists in the iron. I feel that, in working only with the purified graphite extracted from the iron, which the authors have established to be the same as graphite from other sources, they have tended to side-track one most important issue—the nature of the elemental impurities or compounds that may be present in the graphite as it *exists* in cast iron. This information may be of vital importance to explain why graphite occurs sometimes in flake and sometimes in nodular and other forms. Kish graphite has been shown, on chemical analysis, to contain impurities. Very recent electron microscope studies by the Belgian worker de Sy[4] have shown that nodular graphite also often has small inclusions in the centre of each nodule. The nature of these impurities could well be further investigated by X-rays, and conclusion (4) might then require to be modified.

In the paper by Owen it should be recognized that the statement (p. 119) that the solubility of silicon in the carbide phase is negligible must be applied only to irons in the very limited range covered by this research. Dr. Owen (in private correspondence) has agreed to this qualification. It is probable that the position will be made clear on the publication of the results of his present researches, which have been submitted to the Institute, and possibly in his further researches in the U.S.A.

There is one other small point on which further information would be of interest: on p. 118, two references are made to examinations for inclusions in iron–carbon–silicon alloys. What inclusions were expected?

Dr. **A. Taylor** (The Mond Nickel Co., Ltd., Birmingham): I was rather disappointed to find in the paper by Owen and Street that, in spite of all the X-ray work, no information was given about the mechanism of formation of the spheroids or flakes in cast iron. On p. 113 it is stated that the lattice parameter *a* of the β-graphite structure is 2·256 Å.; should this not read $a = 2{\cdot}456$ Å.? I would like to refer to some of the earlier work on the crystal structure of graphite in iron alloys. In 1922, Wever[5] carried out an X-ray investigation of natural graphite and residues obtained by treating several commercial cast irons with nitric acid and with hydrofluoric acid. All these residues were shown to have the same crystalline structure as natural graphite, but the lines of the Debye-Scherrer patterns were somewhat broadened, and did not permit any differentiation to be made between the samples. From the line-broadening, and using the Scherrer formula, the crystallite size was computed to be of the order of 10^{-6} cm. Thus the graphite lamellae in grey cast iron were apparently built up of submicroscopic crystallites. This was rather primitive work, because in 1922 the Debye-Scherrer cameras had not been fully developed and were very small; also, the specimens were rather big for the cameras, so that there would be an apparent broadening of the lines. Modern researches do not show anything like the line-broadening obtained by Wever, and in many cases the crystal size of the flakes is very much larger than Wever supposed.

Reference is made, on p. 116, to 'sandwich compounds' and the work of Trzebiatowski. Owen and Street do not find any change in the lattice parameters; in 1929, however, Konobejewski[6] investigated natural graphite from Ceylon, Mariupol, and America, and also Schungites from Siberia, and concluded, from differences in their Debye-Scherrer patterns, that some graphites contained iron in solid solution. In addition, pure graphite heated at 2000° C. with filings of electrolytic iron gave diffraction patterns with extra reflections, which were interpreted to be superlattice lines, and with a line shift, which indicated a slight change in lattice parameter. Konobejewski interpreted these results to mean that within the lattice of the graphite were formed atomic groupings of iron and carbon with the formula Fe_3C, and the impression was given that graphite from iron–carbon alloys which had been taken, in the molten state, to a very high temperature, always contained iron in solid solution. I tried to repeat Konobejewski's experiments in Newcastle during 1937–38,* using pure Ceylon graphite heated together with Armco iron in a 35-kW. induction furnace; this made alloys weighing about 500 g. The graphite obtained from these iron–carbon alloys gave X-ray patterns with lattice parameters that were always identical with those of a good natural graphite. We found it impossible to get any iron into the graphite lattice, although it is possible to introduce atoms of sodium and potassium between the layer planes of the lattice.[7]

In 1937, Gröber[8] examined cast irons containing 4·05–7·70% of carbon, using both micrographic and

* Not published.

X-ray methods. His objectives were to test Konobejewski's ideas on the solubility of iron in graphite, and to determine whether any real difference existed between primary and secondary graphite. He found that, crystallographically, all the graphite residues obtained by dissolving away the iron in HCl were identical and that, within the limits of experimental error, the lattice parameters were the same as those of natural graphite. This confirmed our own results at Newcastle, and it was therefore concluded that no iron was taken into solid solution. Visible resolution of the high-order K_α doublets was obtained with the graphites extracted by Gröber from high-carbon alloys, whereas only very slight line-broadening, sufficient to prevent resolution of the doublets, was obtained for graphites from low-carbon irons. Experiments were made quenching the iron–carbon alloys from the eutectic temperature, to hinder precipitation along the line *E'S'* of the iron–carbon equilibrium diagram.[8] The slight broadening of the K_α doublets was attributed to the small crystallite size of the secondary graphite. The resolution obtained in the doublets from the hypereutectic iron–carbon alloys was due to the fact that the graphite precipitated in this case was primary graphite.

Gröber, using the polarizing microscope, showed that the flakes of precipitated primary graphite were composed of crystals that were uniformly oriented over their whole length. Alloys with a higher carbon content than cementite, *i.e.*, with 6·76, 7·4, and 7·7% of C, were found to have, in addition to flakes, spherical micro-constituents distributed through the cementite, having the dark colour of graphite and diameters of about 6 microns. Viewing these with crossed Nicols, the spheroids showed a black cross on a light background which did not rotate with the specimen. The appearance was the same as that of the spherulitic graphite described by Schneiderhöhn and Ramdohr[9] and by Ramdohr.[10] The spherulitic graphite is recognizable by a spatial radial directionality of a specific crystal axis. Clearly, only those crystals grow that have their direction of fastest growth normal to the spherical surface.

During an X-ray study of the structure of natural and artificial graphites of high purity, I discovered extra reflections which disappeared on heating the graphite in a mixture of concentrated nitric and sulphuric acids.[11] It was apparent that a periodic disturbance of the lattice was causing the extra reflections to occur, or that some form of dynamic stratification (such as that leading to extra diffuse reflections in diamond and other single crystals) was present. The effect of the acid was to wet-oxidize the structure and to open up the lattice, which, on removal of the acid, collapsed back with the planes rather disordered. Lipson and Stokes[12] showed that a suitable regular displacement of the lattice planes relative to each other could result in the formation of a new unit cell with a *c* axis $\frac{3}{2}$ times longer than normal. The intensities of the X-ray spectra from one of the specimens examined by Lipson and Stokes could be accounted for by postulating 14% of the new β-structure, 6% of disordered structure, and 80% of ordinary α-structure. Nevertheless, there were still a number of outstanding difficulties associated with this solution. More recently,[13] mention has been made of an investigation by Larson of the crystal structure of spheroidal graphite, which is stated to be of the β-form.

The fact that spheroidal graphite gives X-ray spectra showing rather more disorder than flake graphite is not surprising, because the smallness of the crystallites within the spheroids, as revealed by the microscope, and the fact that conditions of crystal growth are hardly conducive to a perfect lattice, would naturally combine to give some line-broadening effects. As the graphitic nature of the spheroids was firmly established by earlier workers, it would have been of great interest if an X-ray investigation could have led to a fuller understanding of the mode of nucleation of the spheroids and the mechanism of their growth.

The natural way in which graphite should grow is by forming a flake, and one would expect the lattice planes to extend rather than to form new curved layers. In recent work in America, a single spheroid of graphite was isolated, mounted at the end of a glass fibre, and skimmed with X-rays, which gradually penetrated the spheroid. The diffraction patterns showed that the spheroid does consist of the curved-layer type of structure, and not a flake type or a radial-plane type. This confirms the observations made with the polarizing microscope.

A number of queries arise concerning the X-ray methods used by Owen and Street. With flat crystals, such as occur in flake graphite, there is a marked tendency for the crystals to align themselves parallel to the axis of the specimen, and all 000*l* reflections would tend to be too strong relative to the others. Thus the amount of β-structure could be seriously in error and not easily comparable with the amount deduced to be present in the spheroidal graphite, which, owing to the spherical crystal shape, is not subject to the effects of preferred orientation. It would be interesting to learn whether a calibration substance to allow for instrument broadening was employed in the determination of the line breadths given in Table III. This Table needs revision, and it would be helpful if rough estimates of the amount of disordered material could be included, so that some correlation with the line breadths could be made. Nevertheless, it is difficult to see how the *amount* of any disordered structure could be obtained by comparing half-intensity *breadths*, because the quantity of material must be proportional to the area under the line contour, and the line breadth is not a measure of this. It is also stated (p. 115) that the ratios of observed to calculated intensities for all the lines of the α- and β-structures were plotted against $\sin^2\theta$, and that two parallel straight-line plots were obtained, but surely this is true only if the *logarithms* of the intensity ratios are employed ?

The conclusion reached in the paper, that the graphite present in iron alloys does not contain any element, other than carbon, in appreciable quantities, seems to be based on lattice parameter measurements. This, of course, can refer only to elements in solution, and does not in any way rule out the presence of foreign material in the form of a nucleus at the centre of each spheroid, which some workers believe to exist. In general, X-ray methods would not be very sensitive for the detection of a small amount of nucleating constituent, and it would be interesting to know whether chemical or spectrographic analyses were carried out to substantiate the statement that the impurities present were negligible.

Mr. **H. Morrogh** (British Cast Iron Research Association) : Referring to the paper by Owen and Street, I would like to endorse the comments made by previous contributors in regard to conclusion (4), p. 116. The conclusion might be more justified if it read "The graphite present in the extract from iron alloys does not contain any element other than carbon," but even then the statement would be open to considerable doubt.

I have carried out chemical analyses on extracts of graphite from cast irons of a wide range of composition, and found that certain impurities were present, such as silica. It could be argued, of course, that the method of extraction is not complete. Reference has been made to the possibility of impurities being in solution or

entrapped interstitially in the graphite spherulites. This may be true ; however, although these graphite spherulites are produced commercially by the deliberate addition of small amounts of elements such as magnesium, cerium, and sulphur, they may also be obtained deliberately when producing alloys of very high purity. Iron–carbon–silicon alloys prepared in the purest form *in vacuo* and then subjected to fairly rapid cooling give spherulitic graphite, so that the presence of impurities is not a necessary condition for the formation of those graphite spherulites.

The nuclei which de Sy[4] claims to show (by electron micrographs) in the centre of graphite spherulites, can be explained geometrically. Consider a spherulite consisting of a number of radially dispersed crystallites with their basal planes at right angles to the radii of the spherulites. In a given microsection, the crystallite at the centre will have its basal plane in the surface of the micro-specimen. Graphite viewed normally to the basal plane is optically different from graphite examined at an angle to that basal plane ; it exhibits a bright metallic reflection, and could easily create the appearance of a nucleus.

Referring to Dr. Owen's paper on the carbide phase in iron–carbon–silicon alloys, I believe that his conclusions are incorrect and are based upon an inadequate appreciation of the metallurgical facts. If the silicon content of eutectiferous iron–carbon–silicon alloys is increased to about 2·5%, the eutectic carbide phase appears to behave as cementite. With more than 2·5% of silicon there appears another phase which metallographically can be distinguished from cementite, and appears to be a ' silico-carbide ' phase. The amount of this phase increases progressively until, at about 7% of silicon, it entirely replaces the original cementite.

The highest silicon content examined by Dr. Owen was 3·14%. An alloy of this composition would contain only a very small proportion of the second silico-carbide phase, and I suggest that Dr. Owen's methods were not sufficiently sensitive to detect this phase.

The author then assumes that the carbide in equilibrium with the liquid at the point L is Fe_3C, and also that, as he is unable to construct the quadrilateral, the point L is at fault. Alloys of the composition represented by the point L would, on solidification, show a very considerable amount—in fact, nearly 100%—of the silico-carbide, and therefore the point C should not be that indicated by the composition Fe_3C.

CORRESPONDENCE

Mr. **A. Allison** (Sheffield) wrote : From the X-ray study of the carbides in white iron, Dr. Owen concludes (p. 119) that the solubility of silicon in the carbide phase is negligible. This sheds new light on the constitution of chilled and white iron. At one time it was thought that the carbide was reasonably pure, comprising a certain amount of manganese and any chromium present in the iron. More recently, studies in graphitization have led to the belief that the white crystals formed on the surface of the iron are actually a complex carbide. It is rather surprising that in a rapidly cooled material like chilled iron the differential freezing should be so rapid.

Since the pearlite also contains carbide, the silicon content of the ferrite in specimen 2 must be greater than 2%, judging by relative areas (*see* Fig. 2 of the paper). In specimen 3, for similar reasons, the pearlite must contain about 6% of silicon. Linear measurement of micrographs of chilled iron indicates that the carbide may be 0·01 in. long and possibly 0·001 in. thick, and Dr. Owen, by passing his carbide through a 200-mesh sieve, apparently confirms these sizes. The minuteness of the crystals may explain the completeness of the separation.

The method of extracting the carbides is similar to that adopted by the late Professor Arnold[14] in his study of the carbides in steel, which in his experiments were pure. In Dr. Owen's paper, chemical analysis of the carbides was probably carried out and would confirm the X-ray analysis.

In the micrography of chilled iron I have noticed that patches of phosphide eutectic are frequently attached to areas of carbide in irons containing 0·35–0·4% of phosphorus ; this indicates the sudden separation of the phosphide eutectic from the carbide.

In chilled iron containing nickel and chromium, if it is considered that the carbide does not contain nickel and silicon, then the ferrite part of the pearlite must be a rich nickel alloy and the chromium contained in the carbide crystals and laminae.

Professor **A. Hultgren** and Mr. **K. Kuo** (Stockholm) wrote : From results, as yet unpublished, of an investigation of the composition of cementite in steels containing various alloying elements, the following data are selected as relevant to the paper by Dr. Owen.

Specimens of a steel containing 0·51% of C, 1·66% of Si, and 0·69% of Mn, were hardened in water from 1050° C. and were tempered for different periods at 500°, 600°, and 700° C., in a lead bath.

The cementite was isolated electrolytically, using 10% HCl as an electrolyte at a current density of 2·5–4·0 amp./sq. in. The carbon contents of the residues varied between 6·7 and 7·0%, indicating almost complete freedom from decomposition of the carbide during electrolysis. The silicon and manganese contents found on analysis of the residues are given in Table *A*.

Assuming that up to about 0·2% of Si in the residue may result from contamination due to silicate inclusions in the steel and, possibly, to some adsorption on the carbide particle surfaces,[15] it may be concluded that :

(1) The silicon content of cementite in equilibrium with ferrite in the temperature range 500–700° C. is nil, or very small.

Table A

ANALYSES OF RESIDUES

Element	Temperature, °C.	Si or Mn, %, after tempering for :						
		3 min.	½ hr.	1 hr.	24 hr.	120 hr.	600 hr.	1200 hr.
Si	500	1·24	...	0·71	0·43	0·36	0·15	0·22
	600	0·59	...	0·13	0·14	0·06	0·20	...
	700	0·13	...	0·07	0·20	0·29	...	...
Mn	700	2·49	3·55	...	4·08	4·28	4·22	...

(2) Cementite formed at an early stage of tempering inherits its alloy content from the martensite (paracementite[16]) and then reacts with the ferrite, gradually reaching its equilibrium composition (orthocementite[16]). At 700° C., this adjustment is remarkably rapid for silicon, being completed in less than 3 min., whereas the manganese adjustment requires about 100 hr.

Dr. Owen's conclusion that silicon in iron–carbon–silicon alloys does not enter the carbide phase, is probably valid (or nearly so) at all temperatures for cementite, when equilibrium is established. Under suitable conditions, however, such as the early stages of cementite formation in tempering martensite and, probably, the early stages of pearlite and bainite transformation, silicon appears to enter the cementite lattice.

Tammann and Ewig[1] found that the presence of up to 5% of silicon and 1–3% of carbon in the alloy has no effect on the position of the Curie point of cementite.

Dr. N. J. Petch (Leeds University) wrote : In a reference to work that I carried out, Dr. Owen might be taken to imply that lines with θ values of 47–51° were used for lattice parameter measurements on cementite ; actually, because CrK_α radiation was used, the lines were at 69–83½°, and so were suitable for sensitive measurements. More information on the accurate determination of the lattice parameters of Dr. Owen's cementites would have been interesting. The present position is that Hume-Rothery, Raynor, and Little have made measurements with CoK_α on cementites extracted from commercial steels ; Dr. Owen has used the same radiation and, although he does not give the actual figures, seems to agree with their lattice parameters. Appreciably different values have been found by Lipson and myself[17] for cementite prepared by decarburizing an oxide, and also in a large number of determinations that I carried out on cementites extracted from pure iron–carbon alloys.[18] Instead of CoK_α radiation, MnK_α[17] or CrK_α[18] radiation was used. My opinion is that these radiations, particularly CrK_α, are more promising for accurate work than is CoK_α, because they give much better films with strong, well-separated high-angle lines that are much easier to measure than are the cobalt lines. Both Hume-Rothery and Owen used the photometric examination of the separation of certain overlapping lines as a check on the accuracy of their values, but I have always found photometers to be disappointingly insensitive to the separation of X-ray lines, and I do not know to what extent this method is an effective check at the level of accuracy involved.

One point relating to Dr. Owen's problem of the solubility of silicon in cementite is the well-known graphitizing action of silicon. Appreciable silicon solubility would be a factor stabilizing against graphitization, since the formation of an alloyed cementite implies that it is more stable than the plain cementite.

AUTHORS' REPLY

Dr. **Owen** and Mr. **Street** (University of Liverpool) wrote in reply : We are grateful to Dr. Taylor for his interesting summary of the papers on the crystal structure of graphite in cast irons, which appeared before his discovery of the extra reflections in the diffraction spectra. The work of Wever and of Gröber was restricted to a much narrower range of microscopic forms of graphite than that on which we have reported, but we are pleased to note that the results are in general agreement with our conclusions. We were particularly interested in the discussion on the question of the solubility of iron in graphite, and thank Dr. Taylor for contributing the results of his hitherto unpublished work in support of our argument.

To explain the difference in the extent of ordering in flake and spheroidal graphite by stating that, in the case of spheroidal graphite, the " conditions of crystal growth are hardly conducive to a perfect lattice," seems to us to be an over-simplification of the problem, since (for example) by annealing white irons it is possible to grow the two forms under conditions that are identical except for a very small difference in the composition.

From a consideration of the geometry of the growth process leading to the formation of a spheroid, it is clear that growth in the naturally preferred direction (*i.e.*, parallel to the closely packed basal planes) is restricted by the interference of neighbouring crystallites. Thus, it may have been predicted that enforced growth in directions normal to the basal planes would result in a somewhat disordered structure. Our results appear to confirm this prediction. However, this effect is a result of spherulitic growth and cannot be considered an explanation of the cause of such growth. Most of the contributors to the discussion have been searching for an explanation of the factors that determine the microscopic form of graphite. This is one of the most important unsolved problems in cast-iron metallurgy, and it seems unreasonable to expect the answer from the type of work reported in these papers. This statement will be more readily accepted if the details of the problem are briefly considered. It is well established that graphite forms by a process of nucleation, diffusion, and growth, and to discover the causes of spherulitic growth or flake formation each of these processes must be examined carefully. It may be that the nature of the nucleus, which may be indigenous or artificial, is the governing factor. We agree with Mr. Morrogh's assessment of de Sy's work on the rôle played by artificial nuclei. The size of a nucleus need be only very small indeed, and we are surprised that Dr. Taylor expected X-ray methods, of the type which we employed, to produce any information about such aggregates. From the work of Darken[19] and Wells, Batz, and Mehl,[20] it appears that the diffusion of carbon in silicon–austenite is not a simple mechanism. However, it is unlikely that this is a controlling factor so far as the form of the graphite is concerned. Information concerning the geometric and crystallographic details of the growth process is fairly extensive, and it is under this heading that our contribution may be classified. Before an adequate theory as to the causes of the various types of growth can be advanced, it is obviously desirable that as much information as possible of this kind should be collected and correlated. Theories concerning the causes, however, should not be confused with descriptions of the effects.

It is suggested that the line-broadening effects could be accounted for, in part, on the basis of small crystallite size. If so, all reflections from spheroidal graphite should be diffuse, the extent of the broadening increasing with the Bragg angle of the reflection. The diffraction pattern from flake graphite should show pronounced broadening of $000l$ lines, but lines such as $10\bar{1}1$ should be relatively unaffected. In the samples we examined, the graphite was fairly well developed. The $11\bar{2}0$ line was sharp on all the films ; when this was compared with the corresponding reflection from a well-crystallized sample of Ceylon graphite, no appreciable broadening was detected. The broadening is given in Table III as a ratio of the width of the $11\bar{2}0$ line, which was used as a basis of comparison. It should be noted that the headings of the last three columns in Table III should read as follows :

	Half-Intensity Breadths ($11\bar{2}0 = 1$)	
$10\bar{1}1$	0004	$11\bar{2}2$

No appreciable general broadening of the reflection from spheroidal graphite samples was noticed. There was some broadening of the 0004 reflections from flake graphite samples, but this was small compared with the broadening of the $10\bar{1}1$ line. It appears, therefore, that broadening due to crystallite size was not a *major* effect.

It was pointed out in the paper that it was " not possible to make an accurate estimate of the amount of disordered structure present . . . " As Dr. Taylor states, the breadth of the $10\bar{1}1$ line is extremely sensitive to disordering, and Table III was included merely to demonstrate that there was some difference in the extent of the disordering in the different samples. In fact, the only conclusion that we drew from the examination of line breadths was purely qualitative—that the flake graphite was less disordered than the spherulitic form.

We appreciate that there is a danger of some error being introduced due to crystals aligning themselves parallel to the axis of the X-ray specimen, thus enhancing the 000*l* reflection. If this happens to a pronounced extent, large deviations from the straight-line plot should be observed, for the 000*l* reflections, when the logarithm of the ratio of the observed to calculated intensities is plotted against $\sin^2\theta$. As is usual with this type of plot, there was some scatter, but we did not find any evidence of abnormally large deviations in the case of the 000*l* points. We conclude, therefore, that the error arising from this source was small compared with the overall experimental error, under the particular conditions of test. (We did, in fact, use a logarithmic scale, and we thank Dr. Taylor for pointing out this slip in the paper.)

No spectrographic analysis of the graphite was performed ; such experimental work would no doubt be very difficult. However, we agree that reliable results of this type would be extremely valuable. Clearly, X-ray work on extracted graphite samples can only detect impurities that may be present in solution in the graphite so that the lattice dimensions are altered. It is unlikely that small inclusions mechanically mixed in the graphite could be detected by X-ray methods.

Dr. Riley suggests that the chemical extraction process removes the impurities from the graphite. It is probably true that mechanically mixed inclusions were dissolved, but we do not believe ' dissolved ' elements were removed by the chemical treatments that we employed. The chemical conditions which lead to a rearrangement of the crystal structure have been described by Taylor and Laidler.[11] The precautions we took to ensure the absence of such reactions are described in our paper. We did obtain a number of diffraction photographs of samples made by powdering small ingots, and these showed the same graphite spacing as in the chemically extracted samples.

We would reassure Mr. Morrogh that we are aware of the existence of an iron–silicon–carbide. Further, we have kept him fully informed of recent work which has enabled us to identify *three* different intermediate constituents which we believe to be iron–silicon–carbides. It seems to us that his assessment of our conclusion is based upon an inadequate appreciation of the theoretical basis of the metastable diagram. Rapid cooling from the liquid is necessary to produce the Marles' carbide[21] which Mr. Morrogh has mentioned ; relative to graphite, under all conditions it is a very unstable ' phase,' and is an unsuitable subject for discussion in terms of the equilibrium diagram. On the other hand, at low silicon contents cementite can be usefully considered as a true metastable phase. There is a large amount of convincing experimental evidence to show that the metastable equilibrium diagram is similar to and associated with the stable iron–silicon–graphite diagram, and the ' double-diagram ' concept has proved very useful. It is true that, as the silicon content of high-purity alloys is increased, more rapid cooling rates are necessary to retain the metastable phase ; nevertheless, since the point *C* will represent the composition of the carbide in the metastable system whatever reaction has caused its formation, it is reasonable to extrapolate data obtained from these alloys to higher silicon contents. We feel that the metastable diagram has become more important since the work of Eash[22] and Morrogh and Williams,[23] who, using alloyed cast irons, demonstrated the formation of undercooled graphite, on slow cooling, by decomposition of a carbide–austenite eutectic. We have extended this experimental work to the study of high-purity vacuum-melted alloys, and have been able to show that the same mechanism of graphite formation applies. X-ray studies have shown the carbide to be cementite. Thus, it seems that *slowly cooled* hypo-eutectic high-purity alloys of high silicon content solidify initially in the metastable system. We chose to carry out lattice parameter measurements on rapidly cooled lower-silicon alloys for two reasons. Firstly, if silicon is capable of entering the cementite structure it seems more probable that it would do so under these conditions ; secondly, a coarser microscopic structure would then be formed, giving clearly defined X-ray patterns. We have discussed more fully elsewhere some of the questions associated with the solidification, in the metastable system, of high-purity alloys.

Although only a few of the samples we examined are described in detail, these do indicate the range of alloys with which we were concerned. The conclusions are intended to apply only to alloys within this range ; however, it appears unlikely that silicon enters the cementite structure in appreciable quantities, even in irons of other compositions. Higher silicon contents result in the formation of one or other of the iron–silicon–carbides mentioned above, whose crystal structures all differ from that of cementite.

We thank Professor Hultgren and his associate for the very interesting confirmatory evidence that they have contributed.

REFERENCES

1. G. TAMMAN and K. EWIG : *Zeitschrift für anorganische und allgemeine Chemie*, 1927, vol. 167, pp. 385–400.
2. O. ZMESKAL and M. COHEN : *Rev. Sci. Instruments*, 1942, vol. 13, pp. 346–348.
3. J. CRANGLE and W. SUCKSMITH : *J. Iron Steel Inst.*, 1951, vol. 168, pp. 141–150.
4. A. L. DE SY : *Metal Progress*, 1951, vol. 59, No. 6, pp. 798–801.
5. F. WEVER : *Mitteilungen aus dem Kaiser-Wilhelm-Institut für Eisenforschung zu Düsseldorf*, 1922, vol. 4, p. 81.
6. S. KONOBEJEWSKI : *Zeitschrift für Kristallographie*, 1929, vol. 72, pp. 381–397.
7. A SCHLEEDE and M. WELLMANN : *Zeitschrift für physikalische Chemie*, 1932, vol. 18. pp. 1–28.
8. H. GRÖBER and H. HANEMANN : *Arch. Eisenhüttenw.*, 1937–38, vol. 11, pp. 199–202.
9. H. SCHNEIDERHÖHN and P. RAMDOHR : " Lehrbuch der Erzmikroskopie," vol. 2, pp. 22–23. Berlin, 1931 : Gebr. Borntraeger.
10. P. RAMDOHR : *Arch. Eisenhüttenw.*, 1927–28, vol. 1, pp. 669–672.
11. A. TAYLOR and D. LAIDLER : *Nature*, 1940, vol. 146, p. 130.
12. H. LIPSON and A. R. STOKES : *Proc. Roy. Soc.*, 1942–43, vol. 181, Series A, pp. 101–105.
13. ANON. : *Metal Progress*, 1950, vol. 58, pp. 729–731.
14. J. O. ARNOLD and A. A. READ : *J. Chem. Soc.*, 1894, vol. 65, pp. 788–801.
15. J. KAMECKI and A. KOTLIŃSKI : *Nature*, 1948, vol. 161, p. 402.

16. A. HULTGREN : *Trans. Amer. Soc. Metals*, 1947, vol. 39, pp. 915–986.
17. H. LIPSON and N. J. PETCH : *J. Iron Steel Inst.*, 1940, No. II, pp. 95P–103P.
18. N. J. PETCH : *Ibid.*, 1944, No. I, pp. 143P–150P.
19. L. S. DARKEN : *Trans. Amer. Inst. Min. Met. Eng.*, 1949, vol. 180, pp. 430–438. *Metals Technology*, 1948, vol. 15, Sept., *Tech. Publ.* No. 2443.
20. C. WELLS, W. BATZ, and R. F. MEHL : *Trans. Amer. Inst. Min. Met. Eng.*, 1950, vol. 188, pp. 553–560. *J. Metals*, 1950, Mar.
21. D. MARLES : *J. Iron Steel Inst.*, 1948, vol. 158, pp. 433–436.
22. J. T. EASH: *Trans. Amer. Found. Soc.*, 1941, vol. 49, pp. 887–906.
23. H. MORROGH and W. J. WILLIAMS : *J. Iron Steel Inst.*, 1947, vol. 155, p. 354.

Joint Discussion on the Papers—

THE PRODUCTION OF HIGH-PURITY IRON ALLOYS ON A 25-lb. SCALE*

By B. E. Hopkins, G. C. H. Jenkins, and H. E. N. Stone

TENSILE AND IMPACT PROPERTIES OF IRON AND SOME IRON ALLOYS OF HIGH PURITY†

By W. P. Rees, B. E. Hopkins, and H. R. Tipler

Mr. **B. E. Hopkins** (National Physical Laboratory) presented the first paper, and Mr. **W. P. Rees** (National Physical Laboratory) the second.

Dr. **M. L. Becker** (British Iron and Steel Research Association) : The authors outline the considerations that led them to adopt a particular standard of iron purity which happens to be distinctly inferior to the best previously achieved at the National Physical Laboratory. The wisdom of this decision is now apparent, for had a higher standard of purity been insisted on in the initial stages, it is extremely doubtful whether comparable results would have been available for publication by now.

The first paper gives a concise description of the air and vacuum melting operations, and particularly of the vacuum melting furnace as it exists today. However, it is not clear why the equipment was made to this design. For anyone contemplating a similar installation it is most important to know what are the essential features, as distinct from those which are still present in the apparatus but which may now be regarded as undesirable or obsolete. Incidentally, no mention is made of the name of the manufacturers or suppliers of the apparatus. I think that public acknowledgement might stimulate the continued interest of those firms that undertake this type of development at considerable trouble and cost.

The vessel appears to be much larger than is necessary, and this is a disadvantage since it frequently has to be evacuated and filled with special gases. The inclusion within it of a circulating pump, a gas purifying train, mechanical tilting devices, water hoses, flexible electrical connections, etc., surely involves undesirable complications, rendering the casing more difficult to keep scrupulously clean and free from leaks.

I have never felt satisfied that the hot-dip sample method for estimating the degree of deoxidation (p. 381) actually shows the state of oxidation of the melt, as distinct from the water-vapour concentration in the hydrogen atmosphere above the melt, but would agree that these quantities are probably sufficiently interdependent. The test itself, however, is interesting, and should be further investigated.

The method of making additions to the melt is adequate for the relatively low alloy contents required in the present work, but if larger or more numerous additions are necessary it seems that an alternative device should be incorporated. I am convinced that the type of equipment described in this paper will become very much more common, not only in the laboratory for research purposes but also as production units ; for this reason, the authors' views on these questions would be very useful.

In the second paper, probably the outstanding feature of the preliminary work is the sharp transition temperature from tough to brittle fracture in the impact tests. It is not understood why this should occur ; nevertheless, I think it is very fortunate, because it enables some precise comparisons to be made between alloys possessing otherwise similar properties.

In the series of melts of high-purity iron (p. 158), the authors have shown clearly that oxygen plays an important part in determining the type of fracture in both the tensile and impact tests. As I understand their results, the presence of more than 0·002–0·003% of oxygen renders the iron liable to show intergranular fracture, instead of cleavage fracture, when broken below the transition temperature. From these results and from the transition curves it is evident that oxygen, probably present as FeO in the lattice, may well be related to the transition temperature, even when the oxygen content is insufficient to promote an obvious intergranular brittle fracture. This conclusion is confirmed by the test results for the iron–manganese and iron–manganese–carbon series where, in general, increasing alloy addition reduces the oxygen content and lowers the transition temperature. On the other hand, in the iron–carbon series (Fig. 18), increasing the carbon content should and did reduce the oxygen content, but actually raised the transition temperature. Although this might appear to contradict the conclusion that oxygen is responsible for raising the transition temperature, it should not be overlooked that in the pure iron–carbon series, where the carbon content is above 0·01%, free iron carbide may take the place of iron oxide in raising the transition temperature. This hypothesis would be confirmed if the transition temperature were lowered by adding a small quantity of a carbide stabilizer such as titanium to a 0·05% C alloy, so as to preclude the presence of iron-carbide films in the normalized material.

Another experiment would be to treat a high-purity iron containing more than 0·003% of oxygen with a small quantity of a powerful deoxidant. If the conclusions I have mentioned are correct, such an alloy should show a lower transition temperature than that appropriate to its oxygen content, as indicated in Fig. 3

* *J. Iron Steel Inst.*, 1951, vol. 168, Aug., pp. 377–383.
† *Ibid.*, vol. 169, Oct., pp. 157–168.

of the paper. Such questions will probably be investigated in later work, but the authors' views at the present stage would be appreciated.

Dr. **N. P. Allen** (National Physical Laboratory): This work is an outcome of a meeting of the Alloy Steels Research Committee held in 1935, at which it was decided that the solution to several problems of the mechanical behaviour of steel required a knowledge of the gas content of steel and of its effect upon the properties. When methods of determining the gas content had been developed, it became necessary to prepare iron sufficiently pure to determine the basic mechanical properties, and pure iron was accordingly prepared in 6-lb. batches at the National Physical Laboratory. The 6-lb. scale, however, proved inadequate, and when the work was resumed at the end of the war it was decided to increase the batches to 25 lb. and to adopt procedures that could, if necessary, be used on the 50-lb. or 100-lb. scale.

A standard of purity had to be chosen. The requirement that every impurity should be below a limit of 0·01 wt.-% was adopted, mainly because this was considered the highest standard that would be practicable. This standard, which some advisers thought too high and others thought too low, proved difficult to attain, but has eventually been consistently achieved and, in some respects, improved on. The choice appears to have been rather fortunate, because recent work on this iron has shown that certain interstitially dissolved impurities exert their most important effects when the quantities added are between 0·002 and 0·02%. The influence of additions of this magnitude can be studied with the iron that is now being produced, although ultimately an even higher standard of purity may be required.

It has been suggested that a considerable quantity of steel could be saved if the yield point of structural steel were raised. In recent years, several high-yield steels have been developed having yield points of about 23 tons/sq. in. They usually contain appreciable additions, totalling about 2%, of manganese, chromium, nickel, copper, or molybdenum. The yield point of pure iron is about 5 tons/sq. in. The importance of small quantities of impurities is illustrated by the fact that the minimum addition that will raise the yield point of pure iron to 23 tons/sq. in. is an addition of 0·04% of carbon—if that carbon is present in the right structural condition. The function of the alloying elements present in high-yield steels seems to be to get the carbon into the right condition, but we do not know yet whether they are being used efficiently for this purpose.

Dr. **W. Steven** (The Mond Nickel Co., Ltd.): The most important feature of these two papers is the careful determination of the tensile and impact properties of pure and alloyed ferrites at different temperatures, and these values will undoubtedly prove useful in future attempts to rationalize the mechanical properties of commercial steels.

An interesting result in the second paper is the susceptibility of occasional batches of the iron to intergranular embrittlement. The authors show clearly that this susceptibility depends on oxygen content; nevertheless, the fact that the iron produced in the 6-lb. high-frequency furnace has a lower tolerance for oxygen than that produced in the 25-lb. furnace, and the behaviour of one or more of the manganese alloys, clearly indicates other important variables. It would seem that there is a critical balance between the amounts of injurious elements present and the amounts of favourable elements present—both amounts being very small—and that minor changes in this balance can cause immunity from or susceptibility to intergranular embrittlement. It is possible that traces of impurities which are important in this balance were derived from the crucible or from the furnace linings. The authors, in discussing the carbon alloys, have referred to the reduction of aluminium from alumina crucibles. It would be interesting to know whether they have also detected traces of magnesium, or possibly of calcium, and have considered the possibility that the presence of these elements tends to neutralize the effect of oxygen. Magnesite linings were used for the first stage of the production of the iron, and the authors mention that although alumina crucibles were used for the second stage the crucibles cracked during the first melt, and that they relied on the backing of magnesite to prevent seepage. From work principally on cast iron[1] it is fairly clear that magnesium can be reduced at high temperatures from magnesite in furnace linings, in amounts much higher than the oxygen content of these irons.

It is difficult to assess the significance of Table X, which deals with the effect of manganese on impact values. Even if the two irons which showed intergranular fracture are neglected and attention is limited to the values quoted for the tempered condition, the results when plotted against manganese content show a wide scatter. There is no more than a trend for transition temperature to decrease with manganese content. The transition temperatures of the carbon and carbon–manganese alloys are more self-consistent, but there are relatively fewer test results and it is surprising that, although carbon alone raises the transition temperature, carbon plus manganese depresses the transition temperature more than manganese alone. The presence of α_2 in the alloys certainly complicates the interpretation of the results, but it is possible that the true effects of the carbon and manganese additions are being masked by variations in the transition temperatures of the base irons. No doubt subsequent work on other alloy additions will clarify the position.

The authors suggest that α_2 may possibly have been present in the 1% and 2% manganese alloys, and this is in accordance with some of the evidence. However, the tempering treatment raised the transition temperatures of these alloys, whereas the same treatment depressed the transition temperatures of the richer manganese alloys, which certainly contained α_2.

A further more detailed study has apparently been made of the effect of oxygen, and this is undoubtedly a valuable line of investigation, as it will not only resolve some of the anomalies apparent in this paper but may throw further light on the embrittlement mechanisms encountered in commercial steel. It is very likely that the type of embrittlement encountered in the N.P.L. iron has much in common with that occurring in commercial steels, but it must be borne in mind that it is possibly wholly characteristic of this very pure material.

Mr. J. Woolman (Brown-Firth Research Laboratories): Dr. Rees suggested that the difference between the 6-lb. and 25-lb. melts might be caused by different rates of cooling in the ingots. Dr. Steven has mentioned the possibility that there are differences in contamination of the steel. I suggest, however, that there may be two simpler explanations of the difference in performance: the amount of reduction from the ingot, which was obviously greater in the one case than the other, and the rolling temperature. I would be interested to know the rolling temperatures used for these ingots, because with this very-high-purity iron the overheating temperature may be exceedingly low. On the other hand, the sulphur contents may be so low that a high

overheating temperature results. It would be interesting to determine the overheating temperatures of these low-sulphur irons, because if a minimum is found in the curve for overheating temperature plotted against sulphur content, it would confirm the precipitation theory we have put forward to explain the phenomenon of overheating.

Mr. **R. D. Carter** (Plessey Co., Ltd., formerly in the Department of Metallurgy, King's College, Newcastle-on-Tyne) : As a result of the present work we were able to carry out an investigation of the occurrence of temper brittleness in iron-base alloys, using the supply of Swedish iron available at the National Physical Laboratory.

We encountered persistent intergranular brittle fracture when examining the behaviour of iron-base alloys containing chromium, manganese, and nickel, in the absence of carbon, and we were fortunate in learning some results of the present work before they were finally published. We confirmed that the presence of oxygen leads to intergranular embrittlement, particularly in the absence of carbon. For example, miniature $\frac{1}{8}$-in. dia. impact test specimens of a 2% Mn alloy, and also of the deoxidized N.P.L. iron, gave a predominantly intercrystalline brittle fracture and a transition temperature of over 200° C. after being treated for 10 days at 720° C. in an atmosphere of hydrogen containing 25% of water vapour. On treating these embrittled specimens at 950° C. in dry hydrogen, the transition temperature was lowered to below — 60° C. and the fracture became entirely cleavage. The specimens were then further treated in wet hydrogen at 720° C. at a lower water-vapour content (15%), and the intergranular brittle fracture returned and was again removed by heating in dry hydrogen at 950° C.

Of the iron-base alloys which we examined, intercrystalline embrittlement was most marked in the nickel alloys. We did not obtain an iron–nickel alloy entirely free from intergranular embrittlement, even after treatment in dry hydrogen at 950° C.

CORRESPONDENCE

Mr. R. Rawlings (Nelson Research Laboratories, English Electric Co.) wrote : The dipstick method of determining the water-vapour content of the hydrogen over the melt is very interesting and obviously fairly accurate. However, there is a very simple method giving more precise information. At the Nelson Research Laboratories we have used a simple type of Regnault hygrometer cooled by solid carbon dioxide and acetone. By this means we obtain an instantaneous figure, and the progress of the deoxidation may be followed continuously. The real usefulness of this method is in giving the trend by a series of readings, so that one can estimate when it ceases to be profitable to hold the melt any longer in an attempt to remove more oxygen.

Mr, **P. Whitaker** (Stewarts and Lloyds, Ltd., Corby) wrote : The Laboratory with which I am connected has for many years produced small vacuum-melted casts of iron alloys for studying the effect of gases on the mechanical properties. Some of the alloys have been remelted under pressure.[2]

In our work we found that arsenic had a very marked effect on the mechanical properties, and I would like to ask the authors if they have analysed their alloys for this element. A small increase in the arsenic content will have the effect of raising the transition temperature.

Using magnesia crucibles under highly reducing conditions, we sometimes experienced a marked pick-up of boron, which may also have a pronounced effect on the mechanical properties.

Dr. C. F. Tipper (Engineering Department, Cambridge University) wrote : There seems to be no doubt, after a study of the evidence presented, that oxygen causes intergranular fracture, but with these low levels of impurities it is unwise to draw too many conclusions about the effect of other variables separately or in combination. There are, in fact, too many variables.

Could the authors please state how they ascertained the degree of intercrystalline as opposed to cleavage fracture ? It would be desirable to establish the distinction on a quantitative basis. If both types of fracture occur simultaneously, the fracture stresses have little significance.

I should like more information about the yields in these pure irons and alloys. Mention of the lower yields in the Tables in the second paper implies that some of them showed an upper yield also. The scale to which Fig. 2 is plotted leaves one in doubt whether there is a genuine yield. The grain size is admittedly large, and it would be interesting to determine the effect of refinement of grain.

In discussing the properties of the carbon–manganese alloys, reference is made to the effect of refinement of grain by the addition of carbon and of manganese. In effect, the tests have been made on material of such different grain sizes that the interpretation of the results is difficult, and I would like to ask the authors if some method could be adopted of controlling grain size without at the same time introducing another variable.

May I express the hope that some of this work will be repeated to check reproducibility ; its value would then be even greater than it is at present.

Dr. **W. I. Pumphrey** and Mr. **W. D. Biggs** (Murex Welding Processes, Ltd.) wrote : Although it has often been suggested that the ductile-to-brittle transition temperature is very largely determined by the oxygen content of the material under test, no definite evidence in support of this suggestion has yet been obtained, and we should be interested to learn from the authors why they have suggested so strongly in the second paper that oxygen promotes intergranular embrittlement in high-purity iron. This suggestion is supported by their results for the irons listed in Table I, but from the results given in Tables V and VI it would seem that with irons 3*AF*1, 5*AF*3/1, and 8*AF*3 a regular decrease in the tendency to intergranular brittleness is accompanied by a regular increase in the sulphur and nitrogen contents, and also by a regular decrease in the carbon and oxygen contents. In fact, on p. 162 it is stated that " the sulphur content of these irons increased in the order of decreasing intercrystalline weakness."

Again, from Tables VIII and X it appears that with certain alloys having approximately equal manganese contents but relatively different oxygen contents (*e.g.*, alloys 27*AF*2 and 29*AF*1 tested in the normalized or normalized and tempered conditions), the ductile to brittle fracture transition temperature is lower in the alloy of higher oxygen content.

We agree with the authors that a low brittle strength would be expected when failure is partly or wholly intergranular, but in the absence of definite evidence of the existence of a grain-boundary constituent, even under the electron microscope (*see* p. 161 of the paper), we feel that a low brittle strength in high-purity iron cannot yet be definitely ascribed to the oxygen content of the iron. We would appreciate the authors' comments on this point.

The effect of grain size is not emphasized in the authors' interpretation of their experimental results, but it is of

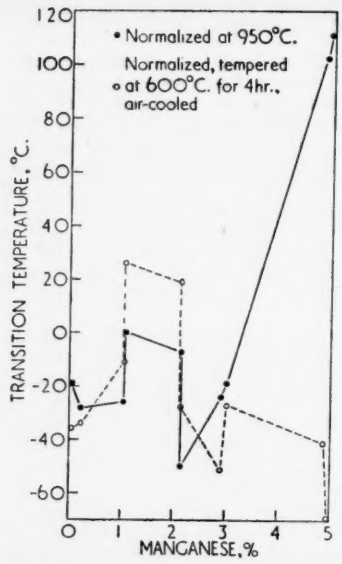

Fig. A—Variation of transition temperature in impact of iron–manganese alloys containing up to 5% of Mn. Tested in the normalized and normalized and tempered conditions

interest to note, as the authors have, that with the two irons containing 1% of manganese (27*AF*1 and 29*AF*2) tested in the normalized condition, the transition temperature observed was lower in the alloy of smaller grain size. A similar result is recorded for the two alloys containing 2·13% of manganese (24*AF*2 and 26*AF*2). The effect of grain size on the ductile to brittle transition temperature has been a subject of investigation for many years, and Davidenkov and Vitman[3] suggested that a reduction in the grain size of a ferrous alloy will always lower this transition temperature. Much of their evidence is inconclusive, but considerable further evidence in support of their suggestion can be found in the literature.

Although the grain sizes of the iron-carbon and iron–carbon–manganese alloys are not given, an examination of the photomicrographs reproduced in the paper suggests that the grain sizes of these alloys were somewhat variable ; did the authors consider the effect of variation in grain size in their assessment of the mechanical properties of these alloys ?

Finally, we suggest that the authors have no conclusive experimental evidence to justify their statement that the effect of an increase in the manganese content of iron–manganese alloys is to decrease the ductile to brittle transition temperature. The results recorded in Table X of the paper are shown plotted in Fig. *A* ; it is apparent that, even if the results for alloys 29*AF*2 (1% of Mn) and 24*AF*2 (2% of Mn) are omitted, the curves are far from smooth and regular, and it would therefore be incorrect to draw a definite conclusion as to the effect of manganese on the ductile to brittle transition temperature. It would seem to be no explanation of the scatter in the results given in Table X to state that certain fractures were intercrystalline and others were cleavage ; such a statement is merely a description of the fracture and is no explanation of the scatter. The scatter is similar to that so often recorded with commercial steels tested under similar conditions, and we would be interested to know whether the authors will be investigating this phenomenon in future work.

AUTHORS' REPLIES

Mr. **Hopkins,** Mr. **Jenkins,** and Mr. **Stone** (National Physical Laboratory) wrote in reply to the discussion on the first paper : Dr. Becker's remark that the purity of the iron now made at the N.P.L. in 25-lb. lots is distinctly inferior to that of iron made earlier on a small scale, needs qualifying. Although the statement is true in some respects (for example, the nickel and copper contents of the present iron are about 0·006% and the manganese content is up to 0·004%), as far as the more important elements carbon, oxygen, and nitrogen are concerned, the best irons made at present are as good as the older N.P.L. Mark 1 iron. In general, however, it is true that to make iron in sufficient quantity for extensive mechanical tests we have tolerated small amounts of certain impurities, mainly metallic, which we do not consider have significant effects on mechanical properties, in such proportions. The sulphur content of our irons is higher than we would like.

The furnaces were made by The Electric Furnace Co., and we would like to take this opportunity of expressing our appreciation of their co-operation. It was understood at the time, however, that once the furnaces had been obtained a considerable amount of laboratory development work would be required before high-purity iron could be produced. The account we have given is of the equipment as it is at present, and we do not think any of the features we introduced in the early stages have become either undesirable or obsolete. The account has concentrated on the essential features.

It is agreed that the tank containing the 25-lb. furnace is large, but we consider this to be a convenience and not a disadvantage, because of the large capacity of the pumping equipment. The subsidiary equipment that Dr. Becker mentions was placed inside the tank purposely, because cleaning presents no problem and this arrangement has the important advantage that a minimum number of joints (which could be sources of leaks) remain outside the tank.

We agree that the dip-stick technique tests the dryness of the hydrogen atmosphere inside the tank, but this is related to the state of deoxidation of the melt. The test has been used mainly as a qualitative one, but we consider that it is capable of further development. In this connection, we are indebted to Mr. Rawlings for his contribution on the use of a Regnault hygrometer, which appears to be an improvement on our technique.

Concerning Dr. Becker's remarks about making additions to the 25-lb. melt, the equipment has so far only been used for additions up to 5%, but the simple method of hanging packets from a cross-piece attached to the rod holding the lid of the furnace has been used successfully for a number of additions to the same melt, by suspending the packets by different lengths of wire so that, if desired, one is added at a time. Obviously, if very large additions have to be made an alternative device will be necessary.

Dr. Steven asked whether we have detected magnesium or calcium in the irons. No magnesium and only a trace of calcium was found in the Swedish base iron. We do not consider that reduction of the oxides of these metals could take place from the fired magnesite lining during the oxidizing melts, and during reduction in the 25-lb. furnace the melt does not come into contact with the fired magnesite packing around the alumina crucible. We should make it clear that, although we stated that cracking of the alumina crucible was of little consequence because the fired magnesite packing prevented the outward seepage of metal, we did not mean that the metal reaches the packing but only that the packing prevents the cracks in the crucible from opening sufficiently to

allow the metal to seep through. If the metal were to reach the packing, it would become contaminated with silicon, by reduction of the small amount of silica present in the fired magnesite. In any case, our carbon contents are traces compared with those of the cast irons that Dr. Steven mentioned. We contend, therefore, that there is no opportunity for these elements to enter the iron. On one occasion when magnesium was added to the iron in the 25-lb. furnace, it was not found possible to retain it ; 0·007% was added immediately before pouring, but none was detected in the ingot. Presumably it had been lost mainly by volatilization.

Mr. **Rees**, Mr. **Hopkins**, and Mr. **Tipler** (National Physical Laboratory) wrote in reply to the discussion on the second paper : We think Dr. Becker would find it difficult to substantiate his suggestion that oxygen present in amounts insufficient to cause intergranular brittleness may be effective in raising the impact transition temperature. All the evidence available at present indicates that this is not so. For example, in the iron–manganese alloys that he mentions, of the pairs of alloys containing 3% and 5% of manganese, none of which shows any sign of intergranular brittleness, the alloy in each pair with the higher oxygen content has the lower transition temperature in impact after heating for 4 hr. at 600° C. They are being compared in this condition to avoid confusion due to α_2 structure.

We would expect that the addition of a strong deoxidizer to iron containing sufficient oxygen to produce intergranular brittleness in the absence of the deoxidizer (more than about 0·003% under our conditions on the 25-lb. scale) might lower the transition temperature by combining with the oxygen and thereby preventing it from embrittling the grain boundaries. We should emphasize that we have no definite microscopic evidence of the presence of iron oxide, as such, at the grain boundaries.

We have not yet carried out the experiment of adding a carbide former to an iron–0·05% carbon alloy, and we feel that at this stage it would be unwise to predict what would happen.

Mr. Woolman suggests that the more pronounced susceptibility to intergranular brittleness of irons cooled slowly in the 6-lb. furnace, compared with the chill-cast irons made in the 25-lb. furnace, might be due to a difference in the amount of subsequent hot reduction. It so happens that the irons cooled in the crucible in the 6-lb. furnace were about $2\frac{1}{2}$ in. in dia., whereas the 25-lb. chill-cast ingots are $2\frac{3}{4}$ in. across the flats of the octagonal section. The difference in the amount of reduction involved in rolling to $\frac{5}{8}$-in. dia. is thus small. In any case, the difference in susceptibility to intergranular brittleness is apparent in the cast condition.

The rolling temperature was given in the paper as 1100° C. We have no information about the overheating temperature of the irons, but we feel it is quite possible that sulphur content would have a marked effect.

In reply to Mr. Carter, we also have found that heating iron in the solid state in moist hydrogen results in severe intergranular brittleness, but we have not carried out the experiment of subsequently heating in dry hydrogen. A paper by Stanley[4] on this subject has since appeared. We agree that susceptibility to intergranular brittleness is affected by alloying elements and that iron–nickel alloys are particularly liable to break along grain boundaries.

We were interested in Mr. Whitaker's remarks. We have looked for boron in the two irons 18*AF*3 and 53*AF*1 by the quinalizarin method of Rudolph and Flickinger,[5] but we did not detect any, so it must be below 0·0003%. The arsenic contents determined by a standard Gutzeit method were 0·001% and 0·0015%, respectively. We think it will be agreed that these amounts are exceedingly small.

In reply to Dr. Tipper, the fractures were inspected under binoculars but the proportions of intergranular and cleavage were not determined. In fact, we do not know of a way of doing this, because, unlike fractures that are partly ductile and partly brittle, in which it is possible to estimate the proportions of the ductile and brittle types since they usually occur in separate areas with a more-or-less sharp line of division, the intergranular and cleavage portions of a brittle fracture are intermixed.

Dr. Tipper is correct in assuming that the irons for which lower yield stresses are reported also generally showed an upper yield, but in some cases there was no drop in load.

It is agreed that in the iron–carbon–manganese alloys the grains are refined by both carbon and manganese, and the assessment of the effect of these elements includes the effect of decreasing grain size. Whereas, for single-phase alloys, it is possible to devise a scheme that will give a similar grain size with increasing content of alloying element, to enable the effect of the alloying element to be determined independently of its effect on grain size, such a treatment applied to two-phase alloys (such as the iron–carbon–manganese alloys), results in other, possibly more important, variations in the alloys. Incidentally, the grain sizes of these particular alloys are not ' variable ' as Dr. Pumphrey and Mr. Biggs state, but they change in a regular manner with increasing carbon and manganese contents.

Dr. Pumphrey and Mr. Biggs agree that the results for the irons made in the 25-lb. furnace enable the conclusion to be drawn that oxygen is effective in promoting intergranular brittleness, but they imply that the results for the irons made in the 6-lb. furnace are not consistent with this. They refer to the increase in sulphur and nitrogen contents in the three irons 3*AF*1, 5*AF*3/1, and 8*AF*3 (Table V) with decreasing tendency to intergranular brittleness. Firstly, the nitrogen contents increase only from 0·0009% to 0·0013% ; this is scarcely a significant difference. Secondly, the sulphur content increases from 0·0016% for iron 3*AF*1 to 0·0053% for iron 8*AF*3, but in spite of this the degree of intergranular brittleness decreases. We feel sure that Dr. Pumphrey and Mr. Biggs do not wish to suggest that an increase in sulphur content is effective in decreasing the tendency to intergranular brittleness. The decrease in carbon content from 0·009% for the first iron to 0·004% and 0·0039% for the other two, should, in the presence of oxygen, increase the tendency to intergranular brittleness (*see* Fast[6]). The reverse actually occurs, and we must therefore conclude that it is the decrease in oxygen content from 0·015% to 0·0007% that is responsible for the diminishing tendency to intergranular brittleness in this series. This agrees with the conclusion drawn from the more complete information available on the irons made in the 25-lb. furnace. More detailed evidence will be published shortly.

Dr. Pumphrey and Mr. Biggs have not appreciated the fact that oxygen seems to affect the properties only when it is present in amounts sufficient to produce fracture partly along the grain boundaries. If the oxygen content is below the level required to produce any intergranular fracture at all, it follows that the grain boundaries are stronger than the cleavage planes, and since the effect of oxygen appears to be concentrated at the grain boundaries, no effect is evident until some sign of grain-boundary fracture is obtained. Their remarks about the 3% Mn alloys 27*AF*2 and 29*AF*1 merely demonstrate this point.

With reference to their insistence on microscopical

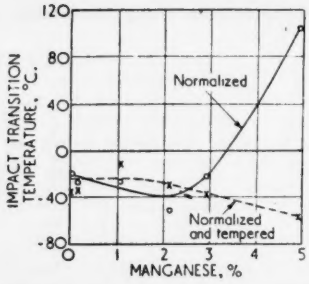

Fig. B—Transition temperatures in impact of iron–manganese alloys tested in the normalized and normalized and tempered conditions

evidence of a grain-boundary constituent before believing that oxygen can produce grain-boundary brittleness in iron, we would remind them that this would only clarify the mechanism of the embrittlement. The fact that the grain boundaries are weakened by oxygen is proved by the tensile tests at — 196° C., for instance. Examples exist of grain-boundary brittleness appearing before there is visible evidence of anything in the grain boundaries; McLean and Northcott[7] found this with antimony in 70 : 30 brass, and McLean[8] has since shown that the same applies to antimony in copper. It is also known that electrolytic polishing is better than hand polishing for revealing grain-boundary effects; *e.g.*, Schofield and Cuckow[9] found this with bismuth in copper. Unfortunately, oxygen in iron is a particularly difficult case, because the reagents used for electrolytic polishing are prone to attack oxide inclusions severely.

Dr. Pumphrey and Mr. Biggs' statement that the effect of grain size on the transition temperature has been the subject of investigation for many years, is true in so far as high impact values at room temperature are often associated with fine-grained material. It is, however, difficult to find evidence of the dependence of transition temperature on grain size alone, because the treatments used to obtain various grain sizes usually result in other variations in the microstructure of the material. For example, in Davidenkov and Vitman's work,[3] the slow cooling incorporated in their heat-treatment at 1100° C. to produce coarse grains in the 0·24% C steel would, in itself, cause a raising of the transition temperature, and the drop in transition temperature of the material when normalized at 950° C. was not necessarily caused by the halving of the grain size that resulted from this treatment.

Dr. Pumphrey and Mr. Biggs have plotted the results of the impact tests on the iron–manganese alloys in a way that exaggerates the inconsistencies. We agree that there is some scatter in the results, but anyone who has had anything to do with this sort of work will agree that the ratio of the two axes in Fig. *A* is unrealistic. The two alloys 29*AF*2 (1% Mn) and 24*AF*2 (2% Mn) do not really belong to the series, for the reasons given in the paper; they were included merely to illustrate the sort of effect that small quantities of certain impurities can have on mechanical properties. We think that Fig. *B*, in which the results are plotted using the average results where there are pairs of alloys of the same manganese content, and omitting the two alloys just mentioned, gives a more balanced picture of the effect of manganese. We maintain that the conclusion drawn in the paper, that the effect of increasing the manganese content is to cause a general lowering of the transition temperature provided that any α_2 formed at the higher manganese levels is tempered, is justified.

REFERENCES

1. H. Morrogh : " Improvements in or relating to the Production of Cast Irons." British Patent Specification No. 654, 911. 1951, July.
2. H. A. Dickie : *J. Iron Steel Inst.*, 1948, vol. 159, pp. 360–375.
3. N. N. Davidenkov and F. Vitman : *Technical Physics of the U.S.S.R.*, 1937, vol. 4, pp. 308–324.
4. J. K. Stanley : " The Embrittlement of Pure Iron in Wet and Dry Hydrogen." *Amer. Soc. Metals, Preprint No.* 15w, 1951.
5. G. A. Rudolph and L. C. Flickinger : *Steel*, 1943, vol. 112, Apr. 5th, p. 114.
6. J. D. Fast : *Metalen*, 1949, pp. 171–181.
7. D. McLean and L. Northcott : *J. Inst. Metals*, 1946, vol. 72, pp. 583–616.
8. D. McLean : Unpublished work.
9. T. H. Schofield and F. W. Cuckow : *J. Inst. Metals*, 1947, vol. 73, pp. 377–384.

Joint Discussion on the Papers—

STRUCTURAL TRANSFORMATIONS IN THE TEMPERING OF HIGH-CARBON MARTENSITIC STEELS*

By K. H Jack

MAGNETIC ANALYSIS OF IRON–CARBON ALLOYS : THE TEMPERING OF MARTENSITE AND RETAINED AUSTENITE†

By J. Crangle and W. Sucksmith

Dr. **W. H. Taylor** presented the first paper on behalf of the author, who was in America.

Mr. **J. Crangle** presented the second paper.

Mr. **E. H. Bucknall** (The Mond Nickel Co., Ltd.) : The papers give very little information about the materials employed. Jack used rods cut from a solidified heat of an iron–carbon alloy in which he inferred that there was 1·3% of carbon, whereas Crangle and Sucksmith

* *J. Iron Steel Inst.*, 1951, vol. 169, Sept., pp. 26–35.
† *Ibid.*, vol. 168, June, pp. 141–150.

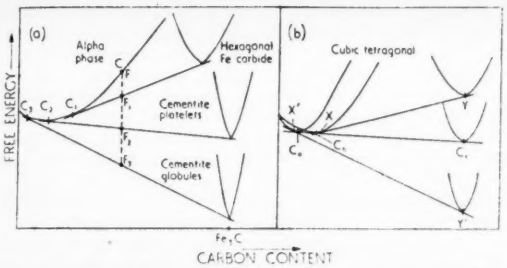

Fig. A—Free-energy/carbon-content curves, showing the various stages of separating phase

state that they prepared alloys by carburizing solid iron, and give the carbon contents of only two of them, although many more were used. Alloys such as those containing 1·3% and 1·7% of carbon usually contain much of the carbon as graphite, but the authors do not seem to have suffered from this difficulty.

Both papers refer to the presence of austenite in the quenched alloys, but only Crangle and Sucksmith indicate the amount, and that only in one of the alloys. It would be of interest to know the estimated austenite contents of their other alloys. If the alloy containing 1·7% of carbon were brought to a homogeneous austenitic condition before quenching, its M_s temperature should be fairly near room temperature and the austenite content should be extremely high. The application of sub-zero treatment to such an alloy, to increase the percentage of martensite, might be worth while.

I would like to know more about the annealing treatment applied by Crangle and Sucksmith to the alloy described in their Fig. 9 as tempered at 808° C. ; why was this treatment expected to give an equilibrium condition ?

Jack states that the three stages of tempering represent : (i) the change of martensite to ferrite, together with the coherent ε-phase ; (ii) the breakdown of residual austenite ; and (iii) the conversion of ε into Fe_3C platelets, and their recrystallization. The first stage is said to be completed at 160° C. and the third at 360° C. Crangle and Sucksmith, on the other hand, can only explain the magnetic properties if the iron per-carbide phase persists to temperatures above 500° C. Is this because their times of tempering were much shorter than those used by Jack, or is there a more fundamental explanation of the apparent disagreement ?

Jack states on p. 32 that ε-carbide ' transforms ' into cementite. I would have expected the process by which cementite is formed to be analogous to that of the θ-phase formation in copper–aluminium alloys, where the transition phase is believed to contain too much copper for $CuAl_2$, and the latter forms as a result of an interfacial reaction with the adjacent matrix.

Jack does not seem to have pointed out that carbonyl iron always contains nitrogen as well as carbon (usually 0·7% of each) and also nickel as an impurity. X-ray work on this type of iron is being carried out in our laboratory by Dr. A. Taylor. Although it is too early to give results, we do not share Jack's belief that carbonyl iron is formed at too low a temperature for it to consist of martensite.

Finally, I would like to refer to the difficulties of nomenclature in this subject. The term ' ε-carbide ' does not have the same meaning in the two papers, and Jack finds it necessary to vary this expression with ε-Fe_2C and ε-Fe_3C. On one occasion (p. 28) we even have FeC_2, which surely should read Fe_2C.

Mr. **D. McLean** (National Physical Laboratory) : The first paper makes fairly clear the structural aspect of the changes that occur when martensite is tempered. Bearing in mind also the work of Clews and Calnan[1] and the recent Russian work,[2–7] it seems that the solutions given in this paper are not likely to be much altered in the future, at least so far as fairly pure alloys of iron and carbon are concerned.

There exists, however, a point of disagreement between the X-ray work and the magnetic work of Crangle and Sucksmith, as given in their paper. Whereas Jack, Clews and Calnan, and also Arbuzov, conclude that the constituent preceding globular Fe_3C is Fe_3C in the form of extremely thin platelets, Crangle and Sucksmith conclude that it is $Fe_{20}C_9$, one reason being that its ferro-magnetic properties differ from those of Fe_3C. Since ferro-magnetism depends on the coupling between atoms, I suggest that the ferro-magnetic behaviour of Fe_3C platelets only about two unit cells thick may differ from that of Fe_3C globules. Moreover, if the final product of tempering is Fe_3C, it seems unlikely that at some earlier stage a phase richer in carbon should form, as it would require more diffusion of carbon.

The Russian workers, whose results are mainly in agreement with Jack's, give certain orientation relationships between the α and carbide phases. These relationships are expressed rather differently from Jack's, and it is not clear whether they are the same ; I would like to know his opinion on this point. In the cementite phase the layers of carbon atoms are parallel to the *ab* plane, and consequently, if the orientation relationship is known, it would suggest the way in which the carbon aggregates in α-iron.

Still unsettled, however, is the other aspect of the changes occurring in martensite during tempering, the elucidation of the structural changes in terms of the interatomic forces. I would like to draw attention to a situation that may occur. Figure *A* (*a*) shows a set of free-energy/carbon-content curves that are consistent with the structural changes described by Jack. As the various stages of separating phase are traversed—hexagonal iron carbide, cementite platelets, cementite globules—the total free energy of the system decreases from F through F_1 and F_2 to F_3, and the carbon content of the iron matrix decreases from C through C_1 and C_2 to C_3.

There should, however, be two curves for the iron phase, one for body-centred cubic iron and one for body-centred tetragonal iron. At zero carbon content the free energies of these two phases are equal, since the *c*/*a* ratio of the tetragonal form approaches unity as the carbon content decreases to zero. For low carbon contents the cubic form is observed, and so is likely to have the lower free energy. At high carbon contents the tetragonal form is usual and so may have the lower free energy. Consequently, it is quite probable that the curves cross as in Fig. *A* (*b*). There thus exists the possibility of the coexistence of the two forms of iron at some stage during the tempering process, the cubic form of low carbon content C_a and the tetragonal form of higher carbon content C_b, since the free energy of a mixture of these two phases for carbon contents of the iron matrix between C_a and C_b is lower than that of either alone. But since the formation of cementite globules (Y', Fig. *A* (*b*)) enables a state of still lower free energy to be reached, the coexistence of two iron phases can only be short-lived. At an early stage of tempering the carbide phase will be in equilibrium with the tetragonal phase, as shown by the tangent $X\,Y$ in Fig. *A* (*b*) ; at a late stage it will be in equilibrium with the cubic phase, as shown by the tangent $X'\,Y'$. At some stage during tempering, therefore, the iron phase

should change discontinuously from tetragonal to cubic, and simultaneously its carbon content should fall discontinuously. This appears to be consistent with the changes that are observed in the iron matrix during the tempering of martensite.

Dr. **D. V. Wilson** (Department of Industrial Metallurgy, University of Birmingham): On p. 35 of his paper, Jack has drawn attention to certain resemblances between the X-ray diffraction effects that accompany the third stage of tempering and those that are observed in a cold-worked steel. In 1942, Andrew and Lee[8] drew some very striking comparisons between the properties of cold-worked steels and those of heat-treated steels, and suggested that there may be similarities in the changes involved in the two processes.

Certain resemblances, of course, are coincidental, and this is probably true of some of the X-ray diffraction effects in the two cases. In a compressed, spheroidized steel cylinder, the cementite diffraction pattern obtained with the X-ray beam perpendicular to the axis of compression[9] shows qualitative similarities to the pattern given by a heat-treated steel in the third stage of tempering. However, the anomalous intensity effects observed with the cold-worked steel are unlikely to be due to the presence of what are effectively two-dimensional platelets of cementite, possibly arising from fine-scale planar fragmentation of the cementite particles during cold working. With the cold-worked steel there is a non-uniform distribution of intensities in the Debye-Scherrer rings, and it appears that, during cold-working, the cementite particles may develop a degree of preferred orientation such that a direction in the neighbourhood of the [001] direction tends to be aligned with the compression axis. In this event a reduced proportion of the crystallographic planes (*hkl*) having high '*l*' indices will be able to reflect in directions perpendicular to the compression axis. Therefore the patterns obtained, when examination is made in this direction, should show resemblances, in intensity distribution, to those obtained under conditions in which the Laue condition for X-ray reflection is partially relaxed in the [001] direction.

On the other hand, the diffraction patterns do suggest that in both tempered steels and cold-worked steels intense textural strains may exist between the ferrite and the cementite particles. These strains may be of the same order of magnitude, but their effects will differ, to the extent that the internal strains derived from cold working will be orientated with respect to the direction of working and will give rise to a macroscopic anisotropy of mechanical properties. It is also possible that in the cold-worked steels, as in hardened and tempered steels, there may be more carbon associated with the strained ferrite structure than is held in solid solution in the fully annealed condition of the steel.

Clearly, further experimental evidence is required, but I suggest it may be these similarities which are underlying certain resemblances in the properties—*e.g.*, certain aspects of the magnetic behaviour—of tempered, quench-hardened steels and cold-worked steels.

Dr. **K. Hoselitz** (Permanent Magnet Association): I was very interested in the authors' remark that they had used the gradient method to prove that during the third tempering stage two distinct carbides can be detected, although the (σ, T) curves at first suggested a heterogeneous mixture of phases with a continuous Curie point range, that is, with continuously varying composition. How sensitive was this method, *i.e.*, how little of a phase could still be detected by a maximum in the $\left(\frac{d\sigma}{dH}, T\right)$ curve? Also, what fields did the authors use for the gradient method?

Another question arises out of our own investigation of a ternary iron–carbon–aluminium alloy. We find that the amount of retained austenite depends very sensitively on the exact quenching speed, and that results on quenched specimens are not very reproducible. Did the quenching method used in the authors' work give reproducible results for alloys with equal composition? Moreover, we find for this ternary alloy that, under certain quenching conditions, a maximum amount of austenite is retained and the second tempering stage is not reached until higher temperatures, sometimes up to about 500° C., are attained. Did the minimum temperature for the second tempering stage, in the pure iron–carbon alloys discussed in the two papers, depend on quenching conditions?

Dr. **J. Nutting** (Department of Metallurgy, University of Cambridge): We have been investigating the decomposition of supersaturated solid solutions of 0·015 wt.-% of carbon in α-iron. After ageing for 1 hr. at 100° C. small precipitates appeared, having diameters of the order of 300–500 Å. Increasing the ageing time resulted in the growth of the precipitates to a plate-like form of dimensions 500 Å. thick and 2000–3000 Å. diameter. After ageing at 200° C. it was possible to identify the precipitates by electron diffraction methods. The pattern obtained was that of the hexagonal iron carbide found by Jack. When the ageing temperature was further increased to 300° C., the microstructure remained the same, *i.e.*, a dispersion of platelike precipitates, but the diffraction pattern showed the presence of cementite (orthorhombic Fe_3C). Thus the pattern of changes found on ageing the iron follows very closely that outlined by Jack for the decomposition of martensite. It is hoped to publish these results in greater detail in the near future.

One point of divergence between the conclusions of Crangle and Sucksmith and those of Jack is in the characterization of the initially formed carbide. If the carbide has the composition Fe_2C as proposed by Crangle and Sucksmith, during the transition to Fe_3C there should be almost a 50% increase in the volume of the precipitated phase. This should be readily detectable by metallographic methods, *e.g.*, carrying out either a lineal or an areal analysis of micrographs from suitably prepared samples. With the iron specimens, no increase in total volume of the precipitates was detected during the transition from the hexagonal to orthorhombic carbide, which suggests that they both have the same composition, *i.e.*, Fe_3C; this is in agreement with Jack's conclusions.

CORRESPONDENCE

Dr. **D. V. Wilson** (Department of Industrial Metallurgy, University of Birmingham) wrote: I would like to have the authors' views on the possibility of an alternative explanation of the results obtained during the third stage of tempering.

After tempering at 300° C., the agreement between the observed intensity values and those calculated for a mixture of ferrite, cementite, and a carbide similar to iron percarbide, is striking (although, if allowance is made for carbon still in solid solution, it appears that the carbon content of the 'unknown' carbide must be significantly less than 8·9%). However, it is stated that attempts to analyse alloys tempered above 300° C., assuming that they consist of mixtures of ferrite, cementite, and this same 'unknown' carbide, are not successful. If this implies, in terms of the present theory, that a complete interpretation of the magnetic results obtained above 300° C. will require the postulation of further intermediate stages in the transformation

of the ' unknown ' carbide to cementite, then the possible influence of elastic strain in the carbide particles should perhaps be reconsidered.

Observations made using cold-worked high-carbon steels have suggested that displacements of the cementite Curie temperature are associated with the presence of internal strains in the cementite particles.[10] Small, hard particles contained in a second solid phase may suffer intense strains as a result of the conditions of their precipitation or as a result of the plastic deformation of the matrix. In these circumstances, apparently, the effects of strain may not be negligible.

Dr. **K. H. Jack** (Department of Chemistry, King's College, Newcastle-upon-Tyne) wrote : The mechanism of martensite tempering proposed by Crangle and Sucksmith on the basis of magnetic measurements is similar to that outlined by Hofer and Cohn,[11] but it differs in at least one important respect from that which I deduced from X-ray diffraction studies.[12, 13] Whereas Crangle and Sucksmith consider that ε-iron carbide transforms to iron percarbide ($Fe_{20}C_9$) during the third tempering stage, I found no evidence for the occurrence of percarbide (Hägg-Fe_2C, according to Hofer and Cohn). The proposed transformations may be summarized as follows :

(i) *Crangle and Sucksmith*

$$\text{Martensite} \rightarrow \underset{\text{carbide}}{\varepsilon\text{-iron}} \rightarrow \underset{\text{percarbide}}{\text{iron}} \rightarrow \text{cementite}$$

(ii) *Jack*

$$\text{Martensite} \rightarrow \underset{\text{carbide}}{\text{coherent and oriented } \varepsilon\text{-iron}} \rightarrow \begin{cases} \text{'two-dimensional' cementite platelets} \\ \rightarrow \text{'three-dimensional' crystalline cementite} \end{cases}$$

Although Crangle and Sucksmith state that Roberts[14] has detected X-ray diffraction lines of iron percarbide in the pattern from residues extracted electrolytically from plain carbon steels tempered at above 250° C., these diffraction patterns have not been published ; they were admittedly vague and may have been confused with those of two-dimensional cementite platelets. Only cementite platelets and possibly ε-iron carbide have been observed by Clews and Calnan[3] in a similar X-ray examination of extracted residues. Since the magnetic results are, as yet, unsupported by X-ray observations, it is important to be assured that the interpretation of the former is unequivocal.

Anomalies which are known to occur [15] in the magnetic analysis of iron–carbon phases and which result from, for example, small crystallite size, unfavourable crystallite orientation, magnetic anisotropy, and strain, may not be entirely resolved by using high field strengths. The effects of strain and crystallite size are particularly relevant to the present problem. The X-ray results[12] indicate that the cementite which is first precipitated during martensite tempering is very highly strained in the [001] direction, and/or the crystallite size in this direction is only a few Ångstroms. Similar observations made by Arbuzov[2] have been reported very recently.[16] From the work of Wilson[10] there seems no doubt that strain caused by cold-work increases the apparent cementite Curie temperature. I have already stated that the X-ray diffraction effects observed in cold-worked steels[17] are similar, except for directional differences, to those shown by cementite platelets during the third steel-tempering stage. If the magnitudes of the strains in the two cases are of the same order, then an increase of 60° C. in the cementite Curie temperature for the platelets is not unlikely. During the third tempering stage, Crangle and Sucksmith observe multiple Curie points between about 210° and 270° C. which they attribute to cementite and percarbide, but on Wilson's evidence the transformations :

$3a_X$: ε-carbide → strained cementite platelets

and $3b_X$: strained cementite platelets → crystalline cementite

are no less plausible than :

$3a_M$: ε-carbide → iron percarbide

and $3b_M$: iron percarbide → cementite,

where the subscripts X and M refer to the X-ray and magnetic investigations, respectively. Completion of the slow change $3b$ is said to be reached in not less than 10 weeks at 400° C. The reaction $3b_M$ of iron percarbide with iron to give cementite is complete after $2\frac{1}{2}$ hr. at 470° C.,[18] whereas the rate of the alternative slower reaction $3b_X$ probably agrees more closely with that observed. Although Crangle and Sucksmith do not record tempering rates or times, it seems unlikely that their specimen tempered at 509° C. (*see* Fig. 8 of their paper) contained undecomposed percarbide, even if specimens tempered at lower temperatures did so. However, according to Isaitchev (*see* Kurdjumov[19]), the lattice strain of precipitated cementite, which is about 0·5% at 300–400° C., persists up to 650° C. It is possible, therefore, that some of the magnetic observations ascribed to iron percarbide may be due to the presence of strained cementite, the existence of which in the tempering sequence is known from X-ray evidence.

The ε-iron carbide gives iron percarbide on thermal decomposition,[20] and Crangle and Sucksmith say there is no reason why the first-stage ε-carbide precipitate in steel should not decompose in the same way. However, Hofer and Cohn give evidence[18] that *both* these iron carbides react with iron to give cementite. Moreover, the reaction of ε-carbide with iron is, under exactly comparable conditions, *more than twice as rapid* as the similar reaction of iron percarbide. The former reaction must be independent of the latter, and might be expected to occur in steels, where a large excess of ferrite is present, in preference to the latter reaction. Some percarbide formation is possible if the quenched specimen is heated rapidly to a tempering temperature at which the ε-iron carbide undergoes thermal decomposition. This condition may apply for some of Crangle and Sucksmith's experiments, but it does not for my X-ray investigation.

The second important feature of the magnetic analysis during the third tempering stage is that the magnetic intensity at temperatures at which the carbide phases contribute little is higher for tempered specimens than for fully annealed specimens. Unless the magnetic properties of strained and unstrained ferrite differ appreciably, the implication that there is more carbide present in the annealed state than in the partly tempered condition is inescapable. This feature may be inferred from the work of Kurdjumov and Lyssak,[19, 21] who find that precipitation of carbide from solid solution continues up to 500° C. The only point of dispute is whether the carbon content of the ferrite at 300° C. can be correlated with the magnetic measurements. At 300° C. not more than 0·1% of carbon (the figure of less than 0·05% used by Crangle and Sucksmith is incorrect) remains in solid solution at equilibrium. This amount may have been exceeded in the experiments under discussion if equilibrium was not established. The composition of the third-stage transition carbide was calculated as 8·8% carbon, assuming that no carbon was dissolved in the ferrite. If, for example, the solubility at 300° C. is 0·1%, then the carbon content of the transition carbide (*not* the average carbon content of the total carbide precipitate) is 7·6%. This composition is within the range I gave for ε-iron carbide. The calculation of magnetic intensities would be modified, of course, if this

carbide had a (σ, T) curve different from that of iron percarbide, and also if the effect of cementite strain was considered. It is therefore not unexpected that quantitative magnetic analyses on the basis of Crangle and Sucksmith's proposals have been self-consistent within only a small temperature range. In view of the possible existence of ε-carbide at the beginning of the third stage, there is no reason why the persistence of ε-iron carbide at 350° C., observed by Heidenreich *et al.*,[22] should be ignored. The material used contained negligible impurity compared with that examined by Hofer and co-workers.[11, 15, 18, 20]

Hofer's experiments are not strictly comparable with either the magnetic study or the X-ray study of steel tempering. Reactions occurring with finely divided mixtures of iron and carbide powders that are initially non-martensitic are not necessarily identical with those that occur in a piece of martensitic steel. In the latter, for example, the first-stage hexagonal iron carbide is formed as a coherent precipitate. Its existence and subsequent transformation are undoubtedly influenced by the considerable coherency strains that exist. Direct chemical analysis shows that Hofer's hexagonal 'Fe_2C' contains not less than 29 at.-% of carbon. It is uncertain that a pure iron carbide of composition Fe_2C has yet been prepared. The ζ-iron nitride Fe_2N has an orthorhombic structure in which the iron-atom arrangement is slightly distorted from close-packed hexagonal.[23] This distortion increases as carbon replaces nitrogen, although ε-iron carbonitrides with interstitial-atom concentrations less than 33 at.-% retain their hexagonal structures.[24] Structural changes in the cobalt–carbon–nitrogen system are similar, and the distortion from hexagonal symmetry is a maximum at the composition Co_2C.[25] An iron carbide with a hexagonal structure would thus be expected to contain less carbon than Fe_3C. The indirect X-ray evidence suggesting that the first-stage transition carbide of tempered steel has a composition between 25 and 30 at.-% of carbon, *i.e.*, nearer Fe_3C than Fe_2C, is given in my paper. If it is Fe_3C, then the Curie temperature calculated from Crangle and Sucksmith's observations is almost identical with that found for Hofer's carbide with a larger carbon content. On the other hand, if the transition carbide composition is near Fe_2C, then the calculated Curie temperature is appreciably smaller than has been observed for a similar composition. Thus, the curve of Fig. 5 (*see* Crangle and Sucksmith) is not in agreement with the limited data available elsewhere, and again it is possible that any anomalies are the result of the high coherency strains which are known to exist in precipitate and matrix. If it is assumed that the first-stage transition carbide is ε-iron carbide of composition near Fe_3C, the changes in specific volume of a 1·3 wt.-% C steel calculated, in my paper, for various stages of steel tempering, are in good agreement with the changes actually observed.[26] The agreement is poor if the transitional phase is ε-Fe_2C, as is shown by the figures at the foot of this page.

Mr. Crangle has examined by magnetic methods a sample of carbonyl iron powder for which I have described the X-ray diffraction effects. Because of the presence of impurities a full phase analysis was not attempted, but the (σ, T) curve was similar to that at the end of the second tempering stage. This agrees with the X-ray interpretation. A similar examination was made of the same powder after tempering to the condition in which the diffraction pattern showed only the presence of strained cementite platelets, and, in spite of this, the full third-stage magnetic effects still occurred. The measured (σ, T) curve was slightly lower at low temperatures than that for the initial state, and no definite cementite Curie point at about 210° C. was observed. Although Crangle emphasizes that both these effects may have been caused by the low experimental accuracy attainable with the small specimens used, the fall in magnetization characteristic of the third stage was shown and the intensity curve tended to approach that of the annealed state at higher temperatures. Martensite and austenite were, of course, absent in the specimens. These observations are evidence that the magnetic effects ascribed by Crangle and Sucksmith to iron percarbide are actually due to the presence of highly strained cementite platelets in which the strain is subsequently relieved by recrystallization. The exact composition of the ε-iron carbide transitional phase is still unsettled, but this uncertainty does not invalidate my X-ray interpretation of the tempering sequence in martensitic steels or my suggested mechanisms for the phase transformations.

Mr. **E. A. Calnan** and Dr. **C. J. B. Clews** (National Physical Laboratory) wrote : There is some controversy between the authors of these two papers as to the detailed structural changes associated with the third stage of tempering. Some work carried out in this Laboratory on the tempering of a 0·6% plain carbon steel (referred to in 1949 at the Institute of Metals Symposium on Metallurgical Applications of the Electron Microscope,[27] and reported in greater detail at the Congrès International de Microscopie Electronique, Paris, in 1950[1]), is of interest in this connection. The specimens used in the electron microscope studies of Trotter and McLean[28] were examined by electron diffraction reflection patterns and by X-ray diffraction of the carbide precipitates extracted electrolytically.

The essential feature is that the plate-like precipitate shown in the electron micrographs of a specimen tempered for 1 hr. at 250° C. was identified by both diffraction techniques as cementite. The modified X-ray intensities reported by Jack were observed, and the hypothesis that these are due to the plate-like shape of the particles was substantiated by the observation of normal intensities in the electron diffraction pattern. The latter was to be expected, owing to the much shorter wavelength associated with the electrons. At higher tempering temperatures the anomalies in X-ray intensities became less marked, until at 450° C. and above the normal cementite pattern was obtained. These observations are, of course, consistent with the electron micrographs and Jack's conclusions. In none of the patterns was there evidence of iron percarbide, $Fe_{20}C_9$.

AUTHORS' REPLIES

Mr. **Crangle** and Dr. **Sucksmith** (Physics Department, Sheffield University) wrote in reply : Dr. Jack's criticism of the mechanism of martensite tempering proposed by us is largely based on his suggestion that certain observed magnetic effects are spurious. In particular, he suggests

Transition	Calculated				Observed[22]
	For ε-Fe_3C		For ε-Fe_2C		
Martensite → ε-carbide + ferrite	−0·5*	−0·4†	−1·7*	−1·4†	−0·6
ε-carbide + ferrite → cementite + ferrite	−1·7*	−1·5†	−0·5*	−0·5†	−1·6
Austenite → ε-carbide	+3·4*	+3·5†	+2·2*	+2·4†	+3·2

* Assumes zero carbon in the ferrite † Assumes approx. 0·1 wt.-% of carbon in the matrix[19, 21]

that one effect is caused by stress raising the Curie point of cementite. This same suggestion also forms the basis of Dr. Wilson's contribution. There is no experimental evidence that attainable stresses can directly influence the Curie point of a ferromagnetic. The effect was sought by Adams and Green[40] in iron and nickel, materials in which there is no possibility of a constitutional change induced by stress. No change greater than the experimental error (± 2° C.) was found at pressures of up to 2200 atm. (about 15 tons/sq. in.). We are not satisfied that Dr. Wilson has, in fact, r roduced any evidence that internal stress does raise the Curie point of cementite. It is not clear what he means by " . . . particles . . . may suffer intense strains," but, unless it is intended that the existence of internal stresses far in excess of the ultimate tensile stress of iron should be inferred, his explanation that an apparent rise of as much as 50° C. in the cementite Curie temperature is caused by internal stress seems most improbable. His published curves show the characteristics which we would ascribe to the presence of an additional phase. We have shown in our paper that cementite formed under conditions similar to those in which Dr. Jack found that cementite platelets occurred first, has the normal Curie point. Dr. Jack states that " . . . some of the magnetic observations ascribed to iron percarbide may be due to the presence of strained cementite," but unless some experimental evidence can be produced to support this hypothesis, it is untenable.

The magnetic phase analysis anomalies described by Hofer and Cohn,[15] which Dr. Jack mentions, are stated by those authors to arise from the use with powdered specimens of an applied field of only 2160 Oersteds. The demagnetizing correction for a powder composed of approximately spherical particles, the magnetic intensity of which is the same as that of cementite, is about 4000 Oersteds. Thus, even with a material of infinite permeability (which the materials under consideration do not nearly approach), Hofer and Cohn could not possibly have reached saturation, and it is certain that their own explanation of the anomalies is the correct one. Our highest applied field was about 17,000 Oersteds, allowing a maximum effective field of about 13,000 Oersteds under these conditions. In connecting the anomalies with our work, Dr. Jack appears to have been unaware of the necessity of correcting for demagnetizing effects in finely powdered specimens.

Further, it cannot be denied that the magnetic evidence shows the presence of a carbide containing more carbon than does cementite ; our heat-treatment times were not shorter than those of Kurdjumov and Lyssak, except for certain treatments at about 500° C. Specimens tempered for short times at 500° C. had always been heated for long enough periods at lower temperatures to be within the treatment of Kurdjumov and Lyssak for removing carbon from solution. Dr. Jack suggests that it is unlikely that our specimen tempered at 509° C. contained undecomposed iron percarbide ; but in his next paragraph he mentions the observation by Hofer and Cohn[18] that at a slightly lower temperature the reaction of ε-carbide with iron is more than twice as rapid as the similar reaction of iron percarbide. These two suggestions appear to be inconsistent, for the second makes the higher carbide present *less and not more* likely to be residual ε-carbide.

Those objections to our proposals that are based on failure to observe diffraction lines from a carbide possessing a complicated diffraction pattern when it is in the form of small particles embedded in a ferrite matrix, cannot be regarded as valid. Dr. Jack found it necessary to temper his specimen at 120° C. for at least five days to detect even the relatively simple pattern of the hexagonal carbide, but there can be no dispute that the first-stage precipitation is effectively complete after less than 1 hr. at 120° C. Dr. Jack had to heat his specimen at 250° C. for six days before the pattern of the platelet cementite could be detected ; but from our magnetic work we know that cementite is present after about 90 min. at 220° C. Also, the electron diffraction photographs of Calnan and Clews cannot be used as a criterion for the presence of iron percarbide, for they were unable to detect any ε-carbide with certainty.

Dr. Jack quotes calculations of specific volume changes in support of his view that the precipitate formed at the first stage is ε-Fe_2C. We consider that the agreement he is able to show with the experimental values is fortuitous, and is a result of certain assumptions as to the amount of carbon remaining in solution in the ferrite at the end of the first stage. Agreement with the experimental specific volume changes for a precipitate containing more carbon than ε-Fe_2C is obtained if Kurdjumov and Lyssak's figure for the amount of carbon remaining in solution after tempering at 170° C. is used.

In our opinion, the magnetic work on carbonyl iron, to which reference is made, supports our view that the cementite platelets are formed before the start of the third stage of tempering. The magnetic evidence would seem to indicate that the tempering treatment for 40 days at 120° C. did not remove very much of the ε-carbide probably present initially, in spite of the platelet cementite being produced. Since this cementite has a form often occurring in primary precipitates, the possibility that it is precipitated directly from martensite that has become depleted in carbon, at a higher temperature than that of the formation of ε-iron carbide from martensite with higher carbon content, merits serious consideration.

We do not deny that Dr. Jack's interpretation of the details of his X-ray observations is substantially correct ; our results do not contradict these. But we do hold that his picture of the general scheme of the whole process contains an important omission, in a direction in which X-ray measurements are insensitive.

In reply to the questions about retained austenite, the amounts observed varied from about 3% in specimens containing 0·7% of carbon to about 50% for specimens containing 1·7% of carbon. The same compositions generally gave about the same retained austenite content (except where a liquid-air treatment was applied) and the second tempering stage always occurred at about the same temperature. There was little variation in quenching conditions between different specimens, however, as the quenching operations always followed the same technique. The specimen compositions derived from the magnetic analysis for the amount of cementite present in the annealed state were always in good agreement with the chemical analysis for the total carbon content. Thus, there was no evidence that any appreciable amounts of graphite were ever present. The annealed state was assumed to have been attained when longer treatments at higher temperatures caused no further constitutional changes, as observed magnetically. No difference was detected in fundamental behaviour between different compositions in the range 0·6–1·7% of carbon, but only in the quantities of the phases involved. The conclusions reached resulted mainly from measurements on specimens with the higher carbon contents.

The gradient method of examining (σ, T) curves that Dr. Hoselitz mentioned, was capable of detecting about 2% of one phase when mixed with another of an appreciably higher Curie point, but by itself it was somewhat less sensitive for Curie points that are close together.

In the 200–300° C. region for the tempered alloys, the $(d\sigma/dT, T)$ curve did not show two separate maxima but a plateau falling off fairly rapidly on either side. This evidence alone does not preclude the presence of a small amount of material with intermediate Curie points, but the curve obtained by calculation from the known properties of the individual phases thought to be present agreed almost exactly with the measured one. In this work the use of field strengths low enough to appreciably reduce the tail effects near the Curie points on the (σ, T) curves so impaired the experimental accuracy that no definite conclusions could be drawn from the curves obtained ; the lowest field that was useful was about 15,000 Oersteds.

Dr. **K. H. Jack** wrote in reply : The results of recent American work presented at the Ninth Annual Pittsburgh Conference on X-ray and Electron Diffraction[29] have confirmed independently the occurrence of ε-iron carbide and of cementite platelets during the martensite tempering sequence, but no positive evidence of the intermediate formation of iron percarbide has yet been given.

Cohen and Roberts[30] report that ε-carbide coexists in metastable equilibrium with a tetragonal martensite containing 0·25% of C, and that both form simultaneously out of the original martensite. Also, the third stage of tempering comprises the formation of cementite at the expense of the carbide, and, concurrently, the low-carbon martensite loses carbon to become body-centred cubic ferrite. Cohen adds that the first-formed cementite is not perfect in structure or in stoichiometry, and temperatures up to 500° C. are required to attain the cementite characteristic of annealed steel. The results of Kurdjumov and Lyssak[19, 21] have therefcre been substantiated.

Results of our recent work[31] indicate that martensite decomposes into ε-carbide and another tetragonal martensite of much lower carbon content (0·5 ± 0·3% C), which is presumably the same as that described by Cohen. We observe only the rather diffuse (101) and (202) reflections from this martensite. Thus, the matrix and precipitate are both highly strained in parallel directions, as might be expected if my proposed formation mechanism is correct. The strain is a minimum in the direction in which the lattice planes of the precipitate and the matrix fit most closely.

The similarity between Arbuzov's results[2] and my own has been noted.[32] Referring to Mr. McLean's discussion on free-energy changes, there is little doubt that the free energies of the iron carbides decrease in the order ε-carbide > iron percarbide > cementite. Each carbide exists in metastable equilibrium with a matrix, the carbon content of which must decrease in the same order. For example, ε-carbide coexists with a tetragonal martensite containing about 0·25% of C, whilst cementite is in equilibrium with ferrite containing almost negligible carbon. Even if the composition of ε-iron carbide is the same as that of cementite, the former will not ' transform ' to the latter without participation of the matrix (*see* Mr. Bucknall's criticism). The structural relationships between ε-carbide and cementite are such that ready nucleation of the latter by the former is possible. However, once some cementite is formed, whether by ' transformation ' of the ε-carbide or by localized decomposition of the 0·25% C matrix to give cementite coherent with the ε-carbide, then the matrix is supersaturated with respect to this cementite and further precipitation must occur. At the same time, the matrix becomes unsaturated with respect to the ε-carbide, which gradually redissolves. In a heterogeneous system, where the formation of a succession of phases *a*, *b*, *c*, *d*, . . . , of decreasing free energy is possible, any precipitate (say *b*) will tend to dissolve if nuclei of a more stable phase (say *c* or *d*) are introduced. Nuclei formation does not depend only on free-energy considerations and, given favourable structural relationships, nuclei of precipitate *d* may appear under certain conditions before those of *c* ; in such a case the precipitate *d* will grow at the expense of *b* without the intermediate formation of *c*. Crangle and Sucksmith (p. 145) state that some cementite is present with ε-carbide at the end of the second stage of tempering. The growth of cementite at the expense of the ε-carbide might then be expected, and because of the presence of cementite any subsequent precipitation of iron percarbide seems unlikely.

Mr. Wilson infers, as I do, that the magnetic effects observed during the third stage of tempering are associated with strained cementite platelets and not with the presence of iron percarbide. An important feature of his demonstration[10] of the apparent displacement of the cementite Curie temperature with cold-working is its mechanical reversibility. Any suggestion that an increase in the apparent Curie point is due to the reaction cementite → iron percarbide + ferrite seems untenable ; otherwise the reaction must proceed in the forward direction during compression and in the reverse direction on ' back-straining.' The changes in the cementite X-ray diffraction pattern observed during cold-working may also be reversible.

Contrary to previous results,[33, 34] Dr. Nutting has found the same sequence of changes on ageing iron containing only 0·015% of C as I observed in martensite tempering. Precipitation of ε-carbide from solid solution should occur only when the matrix carbon content exceeds the value at which it is in metastable equilibrium with the precipitate. If this value is 0·25%, as given by Cohen, then the ageing of ferrite should give direct precipitation of cementite. Similar difficulties arise when the same arguments are applied in the iron–nitrogen system. The decompositions of carbon–martensite and nitrogen–martensite are analogous, and (in the respective systems) ε-iron carbide corresponds with α''-iron nitride ($Fe_{16}N_2$) and cementite corresponds with γ'-iron nitride (Fe_4N). X-ray observations[35] have shown that the first-formed precipitate obtained by ageing supersaturated nitrogen–ferrite (about 0·05% of nitrogen) is the transition α''-nitride, and not the γ'-phase. That is, the same sequence of precipitated phases occurs in the ageing of nitrogen–ferrite as occurs in the tempering of nitrogen–martensite.[36]

In reply to Mr. Bucknall, the sequence of transformations was observed in specimens other than those specifically designated as *A* and *B*. Some specimens, including *B*, were given sub-zero treatment in liquid air, to reduce the amount of retained austenite. The results of more recent work, using 0·7–1·3% C alloys, agree with the earlier results. No graphitization was observed. Unless the initial quench is very rapid, some tempering of the martensite during its preparation is almost unavoidable. However, even slight tempering causes relatively large changes in the intensity ratios of the martensitic pairs of X-ray reflections. An analysis[37] of the X-ray intensity measurements made by Lipson and Parker[38] on carbon–martensite leads to anomalous values for the carbon-atom radius, when compared with the results obtained for nitrogen–martensite. I have suggested[39] that the inconsistencies result from slight tempering of the carbon–martensite specimens during their preparation. In the X-ray work under discussion, only specimens considered to be fully martensitic were used ; they consisted of untempered tetragonal martensite containing less than 30% of retained austenite. The range 260–360° C. for the occurrence of the third tempering stage is quoted from previous workers, who emphasize the time–tem-

perature dependence of the reactions. After 21 days at 350° C. the precipitated platelets of cementite (*see* Fig. 2) still show marked relaxation of the Laue conditions for X-ray reflection. Fully crystalline cementite was obtained near 500° C., in agreement with Crangle and Sucksmith's observations ; for carbonyl iron powder this stage was reached at 350° C., so that time and temperature cannot be the only determining factors.

Mr. Bucknall infers that carbonyl iron consists of martensite. I referred (p. 26) to the work of Hägg and Zener which discredits the existence of so-called 'cubic martensite,' except at low carbon concentrations, where the terms 'ferrite' or 'supersaturated ferrite' are preferred. The name 'martensite' should be restricted to the supersaturated solid solution of carbon and/or nitrogen in iron with a tetragonal structure, and in which the interstitial atoms, being partly ordered, occupy only one of the three sets of octahedral interstices. In a cubic ferrite all octahedral interstices have equal chances of being occupied. As indicated by Mr. McClean, the change from tetragonal to cubic solid solution is discontinuous. There is no evidence that tetragonal iron–carbon martensites containing less than about 0·25% of C have ever been prepared. It is also doubtful whether supersaturated ferrites can be obtained with carbon contents much greater than about 0·025%—the eutectoid composition. Carbonyl iron containing (say) 0·7% of carbon and 0·7% of nitrogen, gives an X-ray diffraction pattern in which the line positions are, within normal limits of experimental accuracy, the same as for ferrite. The existence of a highly supersaturated cubic phase thus seems impossible. Again, for this concentration of interstitial atoms a tetragonal structure would show a separation of at least 4 mm., even for the (002) and (200) reflections ($\theta \simeq 40°$) on a photograph taken with FeK_α radiation using only a 9-cm. dia. camera. The loss in tetragonality of high-carbon martensite to give low-carbon martensite is complete within a few minutes at 120° C. Any martensitic phase in carbonyl iron must be near the minimum possible for a tetragonal structure, and could account for only a small proportion of the carbon and nitrogen present. The remainder must exist as carbide, nitride, or carbonitride. It was for these reasons I stated that the condition of carbonyl iron is similar to that reached by steels at the end of the second stage of tempering.

The term 'ε-carbide' was used to describe the iron carbide precipitated during the first stage, because it is isostructural with ε-iron nitrides and with ε-phase iron carbonitrides. These alloys are homogeneous over a wide range of interstitial-atom concentrations—approximately from $Fe_3(C,N)$ to $Fe_2(C,N)$. To date, no investigation has given unequivocal evidence of the composition of the hexagonal carbide precipitated during the first tempering stage, or of the hexagonal iron carbide formed in Fischer-Tropsch catalysts. These two carbides may be identical, or may belong to the same homogeneous iron–carbon phase without having the same compositions. Crangle and Sucksmith have justifiably assumed that the precipitate detected by magnetic methods during the first tempering stage is an iron carbide of uncertain composition but having a hexagonal structure. If it is accepted that 'ε' is a structural description which does not define a composition, there is no difference in its use in the two papers. On p. 28 of my paper, FeC_2 should, of course, read Fe_2C.

REFERENCES

1. C. J. B. Clews and E. A. Calnan : *Proceedings of the Congrès International de Microscopie Electronique.* 1950, Paris.
2. M. P. Arbuzov : *Doklady Akademii Nauk S.S.S.R.*, 1950, vol. 73, pp. 83–86.
3. Y. A. Bagaryatskii : *Ibid.*, pp. 1161–1164.
4. M. P. Arbuzov : *Ibid.*, pp. 1085–1087.
5. G. V. Kurdjumov, O. P. Maksimova, and T. V. Tagunova : *Ibid.*, pp. 307–310.
6. G. V. Kurdjumov : *Zhurnal Tekhnicheskoy Fiziki*, 1948, vol. 18, pp. 999–1025.
7. G. V. Kurdjumov : *Doklady Akademii Nauk S.S.S.R.*, 1948, vol. 60, pp. 1543–1546.
8. J. H. Andrew and H. Lee : *J. Iron Steel Inst.*, 1942, No. I. pp. 153p–189p.
9. J. H. Andrew, H. Lee, and D. V. Wilson : *Ibid.*, 1950, vol. 165, Fig. 60*c*.
10. D. V. Wilson : *Nature*, 1951, vol. 167, pp. 899–900.
11. L. J. E. Hofer and E. M. Cohn : *Ibid.*, pp. 977–978.
12. K. H. Jack : *Acta Crystallographica*, 1950, vol. 3, pp. 392–393.
13. K. H. Jack : *Australasian Eng.*, 1950, Nov., pp. 55–61.
14. E. C. Roberts : *J. Met.*, 1950, vol. 188, p. 1210.
15. L. J. E. Hofer and E. M. Cohn : *Analy. Chem.* 1950, vol. 22, pp. 907–910.
16. *Metal Progress*, 1951, vol. 59, pp. 878. 880.
17. D. V. Wilson : *J. Iron Steel Inst.*, 1950, vol. 165, pp. 376–381.
18. L. J. E. Hofer and E. M. Cohn : *J. Chem. Phys.*, 1950, vol. 18, pp. 766–767.
19. G. V. Kurdjumov : *J. Sci. Instruments*, 1947, vol. 24, pp. 16–17.
20. L. J. E. Hofer, E. M. Cohn, and W. C. Peebles : *J. Amer. Chem. Soc.*, 1949, vol. 71, pp. 189–195.
21. G. V. Kurdjumov and L. Lyssak : *J. Iron Steel Inst.*, 1947, vol. 156, pp. 29–36.
22. R. D. Heidenreich, L. Sturkey, and H. L. Woods : *J. Appl. Phys.*, 1946, vol. 17, pp. 127–136.
23. K. H. Jack : *Proc. Roy. Soc.*, 1948–49, vol. 195, Series A, pp. 34–40.
24. *Ibid.*, pp. 41–54.
25. J. C. Clarke and K. H. Jack : *Chem. Indust.*, 1951, No. 46, pp. 1004, 1005.
26. S. G. Fletcher and M. Cohen : *Trans. Amer. Soc. Metals*, 1944, vol. 32, pp. 333–357.
27. Symposium on Metallurgical Applications of the Electron Microscope. Monograph and Report Series No. 8, p. 161. London, 1950 : Institute of Metals.
28. J. Trotter and D. McLean : *J. Iron Steel Inst.*, 1949, vol. 163, pp. 9–13.
29. Ninth Annual Pittsburgh Conference on X-ray and Electron Diffraction, 1951, Nov. 29–30, Pittsburgh, U.S.A.
30. M. Cohen and E. C. Roberts : "Martensite Decomposition." *Ibid.*, Pittsburgh, U.S.A.
31. D. R. Curry and K. H. Jack : Unpublished work.
32. K. H. Jack : *Metal Progress*, 1951, vol. 60, Oct., p. 111.
33. L. J. Dijkstra : *J. Met.*, 1949, vol. 1, Mar., pp. 252–260.
34. C. A. Wert : *J. Appl. Phys.*, 1949, vol. 20, pp. 943–949.
35. K. H. Jack and D. Maxwell : Unpublished work.
36. K. H. Jack : *Proc. Roy. Soc.*, 1951, vol. 208, Series A, pp. 216–224.
37. *Ibid.*, pp. 200–215.
38. H. Lipson and A. M. B. Parker : *J. Iron Steel Inst.*, 1944, vol. 149, pp. 123–135.
39. K. H. Jack : "An Interpretation of the Crystal Structures of Carbon and Nitrogen Martensites." Ninth Annual Pittsburgh Conference, 1951, Pittsburgh, U.S.A.
40. L. H. Adams and J. W. Green : *Phil. Mag.*, 1931, vol. 12, pp. 361–380.

ANNUAL GENERAL MEETING, 1951

Joint Discussion on the Papers—

FULL-SCALE BLAST-FURNACE TRIALS* *By J. A. Bond and T. Sanderson*

DISTRIBUTION OF MATERIALS IN THE BLAST-FURNACE. Part III—FURTHER FACTORS INFLUENCING THE DISTRIBUTION OF SOLIDS IN THE BLAST-FURNACE†

By R. Wild

(*The discussion on the other papers presented at the Meeting was published in the February, 1952, issue of the Journal*)

Mr. **T. Sanderson** (Workington Iron and Steel Co.) presented the first paper, and Dr. **R. Wild** the second.

Mr. **J. H. Patchett** (Dorman, Long and Co., Ltd.): It is now generally accepted that the elimination of fines and large lumps of material from the blast-furnace gives more economical and smoother furnace operation. This results in a lower coke rate and therefore greater output and better and more regular quality of iron, as well as less loss of iron in the form of flue dust. It is still not known how much the output of iron can be increased beyond that automatically obtained by the reduction in coke rate, without sacrificing some of the improvement in coke rate.

In Table II of the paper by Bond and Sanderson, the substantial increases in output which were obtained in No. 9 and No. 10 furnaces, with the good burden and Appleby-Frodingham coke, may have been gained at some sacrifice to the improvement in coke rate which might result from a more restricted blowing rate. Under the above conditions, No. 9 furnace gave an increase in output of 28% and a reduction in coke rate of 3·9%, and No. 10 furnace gave an increase in output of 45% and a reduction in coke rate of 1·9%. When a comparison was made of normal and dried burdens (Table IV), No. 5 furnace showed an increase of 30% in output with an improvement in coke rate of 9·7%, and No. 6 furnace gave an increase of 14% in output and a reduction in coke rate of 2·2%. These results show that substantial saving in coke consumption and improvement in output can be obtained by full preparation of the burden, and this is confirmed by my own experience on home and foreign ore burdens. It appears that improvement in output beyond a certain point can be obtained only by the sacrifice of improvement in coke rate.

The paper refers only to results obtained by additional drying and screening of Lincolnshire and Northants ores; could the authors state the total improvement to be expected from the crushing, screening, drying, and sintering of the furnace burden at the Appleby-Frodingham works? Much of the benefit gained from ore preparation can be lost as a result of inferior coke quality and excessive amounts of smalls charged into the furnace. Further proof of this is given by the results obtained with varying cokes and the addition of Welsh coal in the oven mixture. The ideal fuel appears to be a fast-burning coke with a size range of 1¼–3 in. It is not clear how the marked improvement in the Workington trials, which showed a drop in the coke rate from 19·44 cwt. to 16·88 cwt. during the last two years, was obtained, but I have no doubt it was mainly due to the ore preparation and to the size-grading of the coke.

In the Cleveland district satisfactory results can be obtained by using raw Cleveland ironstone, provided that it is crushed and screened. The burden is 25% raw Cleveland ironstone, 25% sinter, 25% Swedish ore, and 25% a mixture of African ores, all fully screened, excluding ¼-in. material. In comparison with similar burdens unprepared, the saving in coke is about 2 cwt./ton and the improvement in iron output is 12–20%. This seems a very big variation, and I believe that an even greater increase in output could be obtained, although at some sacrifice of the improvement in the coke rate.

The economics of preparing a burden on foreign-ore practice can be based mainly on the cost of sintering or other methods of rendering the fines fit to be charged into the blast-furnace; the cost of crushing and screening is only a small part of the cost of conveying the burden from the stockpile or truck to the furnace skip.

The bulk preparation of the furnace burden, screening out fine material, is therefore fully justified; in addition, the improvement in the blast-furnace performance enables the steel plant to obtain a very much better result per furnace, owing to the better and steadier quality of the iron delivered to it. This is particularly the case with British basic open-hearth practice, because the home ores are usually rich in phosphorus, and the reduction of their phosphorus contents to below 1% is extremely difficult. To reduce the metallurgical load on the steel furnace to a reasonable figure, it is necessary to deliver iron containing not more than 0·8% of silicon and as little sulphur as possible from the blast-furnace.

It has been stated that high-pressure blowing will obviate the necessity of burden preparation, by producing the same benefits in furnace operation. I do not accept this view. The evidence we have is that some improvement in output can be expected, but only slight improvement in coke rate. I consider that the reduction in coke rate which can be obtained by burden preparation is of great value, and that we should aim at the highest possible Rice rating.

Referring to Dr. Wild's paper, no hard-and-fast rule exists regarding the distribution conditions that give the best results in furnace operation. The requirements vary from time to time, and are governed by other conditions in the furnace stack. It is important, however, that the furnace operators should have the means of varying the conditions at the charging level; previous work by Dr. Saunders and Dr. Wild and this latest work give the operators some valuable information.

Mr. **R. P. Towndrow** (Colvilles, Ltd.): Referring to Experiment (1) in the paper by Bond and Sanderson, it is appreciated that the statistical conclusions in Table III are obtained by the application of standard methods of analysis to the results in Table II. Nevertheless, the results must also be regarded from a purely visual point of view, and on this basis I find it difficult to agree with all the conclusions reached.

The conditions referred to as 'normal' do not appear

* *J. Iron Steel Inst.*, 1951, vol. 168, May, pp. 24–39.
† *Ibid.*, 1950, vol. 166, Dec., pp. 339–348.

to be reproducible with any greater accuracy than some of the differences which are being looked for as a result of burden changes. It is a pity that more details of the operating conditions are not available. For example, what was the scrap usage ? What was the flue-dust production ? What changes in slag volume were brought about by the changes in the burden ? The coke rates throughout the experiments were substantially constant, and it would therefore be interesting to know if the wind blown was adjusted to maintain a constant coke rate ; *i.e.*, was the wind reduced very considerably in conditions of poor burden and purchased coke, to prevent the coke rate from rising too rapidly ?

The order of arrangement of the results renders comparison a little difficult, but we are presumably asked to accept that the two furnaces could be regarded as interchangeable, since all combinations of burden and coke were not tried on each furnace. When the results for the combinations tried on both furnaces are averaged, it is clear that the results obtained with a good burden on both normal and purchased coke are very little different from those obtained with a normal burden and normal coke. It is only when the good burden is combined with the Appleby-Frodingham coke that an outstanding increase in production is obtained. Similarly, the poor burden with Appleby-Frodingham coke gives better results than normal burden and coke, and it is only when poor burden is combined with purchased coke that there is a marked decrease in output. When the poor burden is used with the normal coke, the results are very similar to the normal practice. It must be concluded, therefore, that the overriding factor in the experiments was not the quality of the burden but the quality of the coke ; it is a pity that an opportunity could not be found for a run with normal burden and Appleby-Frodingham coke.

The first three conclusions given in Table III seem to exaggerate the effect of the burden and to diminish that of the coke. To confirm these results it would be highly desirable to repeat the entire series. Even if the results were not duplicated on an absolute scale, it could be seen where the ' differences ' were reproduced quantitatively.

A little more explanation would be valuable in the discussion of the results of Experiment (5). It is stated that Thurcroft slacks were coked with a " longer carbonizing time " ; this should surely read " shorter carbonizing time " (20 hr. at Brookhouse, 35 hr. at Thurcroft). The only test in which coke *A* was used was five weeks on No. 1 furnace ; in this period the coke consumption was the lowest for all tests on this furnace, and the output was the second highest. It is difficult to see, therefore, how the conclusion in the final paragraph, that " the fast rate of carbonization, coupled with a high temperature and narrow oven, produced a coke which had an inferior blast-furnace performance ", is reached ; the only test in which Thurcroft coal was carbonized under such conditions appeared to produce results better, if anything, than the normal practice using mixtures of Brookhouse and Orgreave coke. On the other hand, performance certainly appears to be improved when using coke made from the Brookhouse slack carbonized in the Thurcroft ovens. These two circumstances appear to be contradictory. The extent to which the widely varying proportions of sinter might influence the results should be indicated.

In the experiment at Workington on blending a low-volatile coal, results for a one-week test period are shown, unfortunately without any data for the other operating variables of the blast-furnace. This not very convincing result is supplemented by the statement that figures available over a longer period showed improvements upon the test week data ; these figures would give valuable information.

It would be interesting to hear the results of the projected two weeks' trial experiments on breeze blending. Published information on breeze blending and its effects on the blast-furnace is conflicting, and it would be dangerous to draw any conclusions which were not fully supported by adequate experience in the furnaces.

I am surprised that the data on the percentage of sinter in the charge have not been analysed statistically, but have simply been presented graphically. If the coke rate is plotted against the sinter percentage, there is every appearance of a strong correlation. Correlation, however, appears to be even stronger if the coke rate is plotted against the slag volume, and I suggest that the changes in coke rate have, in fact, been produced primarily by changes in slag volume. There is also a certain amount of correlation between sinter percentage and slag volume, and it is therefore not surprising that there appears to be a correlation between sinter percentage and coke rate. Nevertheless, I feel strongly that these two variables are only related at second hand.

In the experiment, the sinter was increased equally at the expense of hard and soft ores ; this was probably the best method if, as seems likely, the reducibility of the sinter is about half way between that of the hard and soft ores. The most remarkable feature in this and the succeeding experiment on coke-burning rate, is the very low blast pressure required for such a large wind volume. In the coke-burning experiment, wind volumes of 61·5 and 57·4 cu. ft. per lb. of coke are recorded. At Clyde Iron Works, on a 20-ft. furnace, the maximum recorded tonnages of coke burnt in one week are 3953 and 3928. In this case, however, blast volumes of less than 42,000 cu. ft. were required, and in our practice it is seldom that figures above 52 cu. ft. of wind per lb. of coke are recorded. Even bearing in mind the probable errors in the metering of blast at both works, the above figures seem to indicate a very much larger solution loss in the Clyde Iron Works practice as compared with the Workington practice.

Mr. **A. Stirling** (Stewarts and Lloyds, Ltd.) : Referring to Table XX of the paper by Bond and Sanderson, the weight of the total burden should be included. By deducting the slag volume from the burden, a useful coke-rate regression formula can be obtained :

$$Q = aV + bB_{fV} + c \pm d$$

where Q = coke rate, lb./ton of iron
V = slag volume, lb./ton of iron
B_{fV} = burden free of slag volume
a, b = regression coefficients
c = regression constant
d = possible error.

At Corby, under the following conditions :

	Furnace 1	2	3	4
No. of weeks in sample for regression analysis	87	87	87	34
Month of campaign :				
at start	27th	19th	8th	1st
at finish	46th	38th	27th	8th
Average conditions :				
Iron, tons/day	461	372	483	450
Slag volume, lb./ton	1872	1906	1815	1853
Burden free of slag volume, lb./ton	4924	5111	4765	4881
Coke rate, lb./ton	2191	2283	2108	2206

the regression coefficients and regression constants obtained were :

	Furnace 1	2	3	4
a	0·3813	0·5268	0·5698	0·4270
b	0·2012	0·0867	0·0494	0·1493
c	486	836	839	686

This form of regression analysis is being continued.

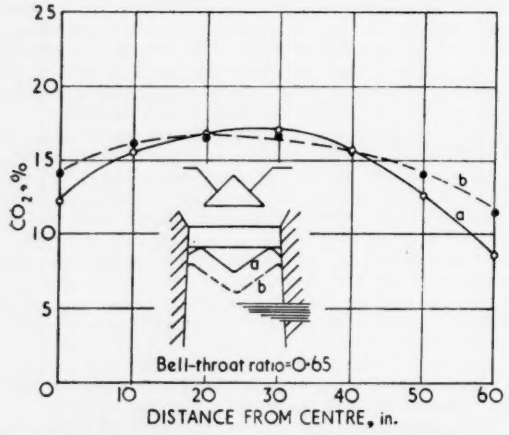

Fig. A—Top arrangement and CO_2 distribution at various stockline heights in blast-furnace No. 5 at Domnarfvet

Mr. **E. H. Baldwin** (Colvilles, Ltd.) : In the section of their paper on sinter trials, Bond and Sanderson have used their very complete data, for each of 19 weeks of furnace operation, only to obtain a series of average values, which they correct (p. 37) with unspecified constants derived from another source. An alternative method of interpretation, which makes the fullest possible use of the 19 weeks' data by using them to construct a regression equation for the coke rate over the period, is, I think, more accurate. Although 19 weeks is a short period for the derivation of an equation, the same criticism may be made of any information derived directly or indirectly from the trial ; also, the constants so obtained have the advantage of being directly related to the behaviour of the furnace at that time. The equation I have obtained is :

$$\text{Coke rate, cwt./ton} = 15{\cdot}07 + 0{\cdot}6366 \times \text{slag volume} - 0{\cdot}007963 \times (\%\ \text{sinter}) + 0{\cdot}03339 \times (\%\ \text{soft ores}) - 0{\cdot}005062 \times \text{blast temperature, °C.}$$

The fourth significant figures are not important. This equation has a correlation coefficient of 0·91 and a standard error of 0·23 cwt. The goodness of fit would have been much better if the scrap and scale had been considered ; these appear to be responsible for the tendency of the calculated coke rate to be lower than the actual coke rate before the 3-week period of high sinter, and higher after the 3-week period. The fact that these differences are constantly maintained during the latter period indicates that they are not due to a persistence of beneficial effects from the high sinter period.

It should be emphasized that, with the given data, no other method of approach can yield more accurate results. The slag-volume coefficient is higher than would be expected on purely thermal considerations, but in my experience this usually happens when no other factor, such as the percentage of iron in the burden, is used to express the richness of the charge.

The equation shows that the lower coke rate obtained by increasing the sinter in the burden is appreciable only when sinter replaces soft ore. For example, comparing the 3-week trial period with the average of the previous and subsequent periods, the sinter during the trial period was increased by roughly 22%, whereas the hard and soft ores were decreased by 10% and 12%, respectively. Substituting 10% of sinter for 10% of hard ores in the equation decreases the coke rate by only 0·08 cwt., whereas the substitution of 12% of sinter for 12% of soft ore decreases it by 0·5 cwt. This point is interesting, because it shows that the effect of sinter is that of a rather good hard ore. The foregoing is valid only if the substitutions involved have little effect on the slag volume. The correlation between slag volume and burden constituents as listed in Table XX is not significant, and a better approach might be to make use of the chemical analysis of the ores. Can the authors state what relationships they have found between slag volume and burden constituents, and whether any provision is made for this in the corrections which they have applied ? It would not be appropriate to correct to equal slag volumes if the use of high percentages of sinter involved an increase or decrease in slag volume.

Mr. **C. Danielsson** (Domnarfvet Iron and Steel Works, Sweden) : Figure *A* shows some results obtained by taking gas samples in our furnace at Domnarfvet ; this has a hearth diameter of 11½ ft. and a throat diameter of 10½ ft. We installed a gas sampling device of German design (the same type, I believe, as that used in England by B.I.S.R.A.) to investigate the distribution of the gas, which must correspond to the distribution of fine and coarse material. It was not possible to make many changes of conditions in the furnace (working with 100% sinter, with limestone mixed in the sintering plant, and with very simple charging devices), but we thought that it would be of interest to find the most suitable stockline for that particular furnace. The normal stockline is shown by the dotted line.

We found that when the stockline rod was shortened from 10 ft. to 8 ft., the CO_2 distribution in the furnace changed to curve *a* (the normal curve *b* is shown dotted) ; the average CO_2 is higher for curve *b*, *i.e.*, the results are better for the original stockline. The figures are of particular interest only to ourselves, but I should point out that the CO_2 content is rather high, the average being about 16% for the whole furnace. This is because we use a high percentage of sinter, a rather high Fe-content (55–56%) of the sinter, and because we grade the coke and the sinter. If materials of different densities are mixed they will always separate if they are of different grading, but if they are of the same grading they will never separate again.

This is probaby what is happening in the furnace at Domnarfvet. We grade the sinter into different sizes from ¼ in. to 2 in. and from 2 in. to 4 in., and the coke from about 1 in. to 2 in. and from 2 in. to 4 in. By using this burden and only sintered concentrates, and by bedding the concentrates, we obtain very uniform material in the furnace. Next year we hope to bed the coke also, and to obtain a further decrease in coke consumption.

Mr. **A. T. Ledgard** (Dorman Long and Co., Ltd.) : Operation may be affected by coke quality in a certain locality or by imported ores, over which those responsible for the furnace have no control, but the use of well-designed and well-maintained furnace charging gear is a factor, under our own control, which more than any other variable can influence furnace operation.

I am in full agreement with Dr. Wild in regard to the quick flow of materials from the bell, and also, in general, about the rotation of the hopper ; but one point he has missed is the discharge of material from the skip itself. To avoid the segregation of fines in the hopper, the speed of tipping and the angle of tipping must be adequate or materials will dribble on to the receiving hopper and so into the rotating top.

The very marked effect of charging sequence on furnace operation was illustrated by one of our furnaces, for which the charging sequence was CC/OO/CC/OO. The coke was in 40-cwt. loads. Trouble from top hanging was continuous for weeks and months, and the filling

was therefore changed to CO/CO/CO/CO, still keeping the skip at 40 cwt. of coke. During the following week the top hanging slowly disappeared, but after six days there was very severe bottom hanging. We therefore tried to compromise, and reverted to CC/OO/CC/OO, changing the skip from 40 to 30 cwt. of coke ; at once the furnace drove freely. This method is still being used.

Recently we had a very similar occurrence in another furnace, in which the mixed filling was OCOC/OCOC. This resulted in bottom hanging for three or four hours at a time, and fairly frequently, so the filling was changed to CCOO/CCOO. This stopped bottom hanging, but resulted in top hanging, so we then used a filling OCOC/OCC, which was very popular at Redcar for a considerable period.

It was thought that the filling OCOC/OCOC, which resulted in bottom hanging, would counteract the filling that gave top hanging. Results were obtained in about 36 hr. ; there was no slipping, the furnace drove freely, and a good output was obtained with a reasonable coke consumption. In a week, however, the whole of the furnace stockline in the region of the lining was in a constant state of agitation, the materials being thrown up to a height of 6–10 ft. This was remedied when the filling was changed to OCOC/OCC–O/, the effect being noticeable within 10 rounds, *i.e.*, just under half a furnace filling. We used a seven-skip round with eight-skip dumping, so that the next round became COCO/CC–OC. This gave the ideal filling, because the materials progressed across the stockline and also vertically in the hopper.

With regard to the **M** or **V** stockline, the furnace is the deciding factor ; if there is rapid movement at the furnace walls, an **M** stockline will soon flatten out and a **V** stockline could finish up in an inverted form. More information on stockline exploration is therefore required.

With reference to the paper by Bond and Sanderson, Neustaetter stated, in a summary of cases of outstanding blast-furnace operation,* that the existence of only one favourable condition, such as low slag volume, good structure of ore and coke, or high top-pressure, will not lead to record production ; a number of favourable factors is necessary to achieve the best results. On the other hand, only one unfavourable factor is enough to produce poor results. This is confirmed by Bond and Sanderson's first experiment (p. 24) on the effect of selected cokes and graded burdens. In Table II, their iron output with a good burden and a poor coke is 3220 tons ; with a good coke and poor burden it is 3060 tons ; and with a good coke and good burden it is 4190 tons.

In regard to the investigation relating to the Rice formula (p. 39), the figure of 3900 tons of coke burned, representing 84·4% of the Rice rate, could surely have been improved by decreasing the temperature from 1250° to 1000° F. As is well-known, lowering the blast temperature increases the concentration of oxygen, enabling more coke to be burnt at the expense of the coke rate.

Mr. **B. G. N. Fowler,** (Appleby-Frodingham Steel Co.) : The authors point out that large-scale trials require " careful planning and subsequent interpretation in the light of statistics." For the statistician to play his part most efficiently, the following three points at least are fundamental :

(i) He should know explicitly the object of a trial
(ii) He should be concerned with planning the trial
(iii) He should be supplied with accurate data.

The first requirement may be thought to be obvious ; for example, in the trial on selected cokes and burdens at Appleby-Frodingham (p. 24) it might have been said that the object was to obtain all possible information on the effect of graded burden and selected cokes. It must be emphasized, however, that any experiment is of limited extent, and can give only a limited amount of information. In this trial many questions might be asked. For example, what is the effect of normal versus poor burden, and of good versus normal burden ? Are these effects the same for both furnaces, and are they the same for different cokes ?

There are 17 questions of this type, and it would clearly be impracticable to attempt to answer them all in a single relatively small trial. One must, in fact, decide on those questions which are most important and design the experiment to answer those only. This does not mean that one must ignore other results of the experiment if they are apparent, nor does it mean that these other questions are not considered. One of the objects of designing experiments is to allow for the possibility of factors other than those in which one is directly interested, affecting the results.

The need for accurate data is obvious, but it is not so obvious how to obtain them. For example, if heterogeneous types of ore are being charged, it is difficult to estimate the chemical and physical properties of the burden. At Appleby-Frodingham we have developed extensive sampling schemes, and we are confident that the data are very accurate ; but it should be stressed that these sampling schemes have resulted from a great deal of experimental work, in which statistics has proved useful and the three fundamentals (i)–(iii) have been constantly observed.

AUTHORS' REPLIES

Dr. **R. Wild** said in reply : Mr. Danielsson's results for the change in CO_2 content across the stack when the stockline contour was altered from a **V** to an **M** contour, are in general agreement with the change in distribution of solid materials which is expected to result from this contour change. The practice of layer charging according to size, as carried out at Domnarfvet, certainly minimizes the segregation caused by size differences, but the problem of segregation resulting from differences in density still remains.

Mr. Ledgard's remarks on the differences in furnace behaviour which result from alteration of the ore/coke cycle show that this variable may have an important effect, and may be used to control the solid distribution across the stockline. I am in full agreement with him that the way in which a furnace is driving has an important effect upon the stockline, because the distribution of gas through the burden depends upon the interaction of two factors : the initial distribution of gas from the tuyeres, and the distribution of solid materials from the bell. The more information that can be obtained on the stockline contour of driving furnaces (particularly of the change in contour between charges), the more accurate will be the overall picture of the effect of these factors upon the distribution of gas across the furnace.

Mr. **Bond** (Appleby-Frodingham Steel Company) and Mr. **Sanderson** (Workington Iron and Steel Company) wrote in reply : We probably did sacrifice some coke-saving by blowing the furnaces for high outputs during the test periods. But calculation shows that to make 4190 tons of iron at No. 9 furnace with the normal blowing rate, the coke rate would have had to be down to 18·1 cwt.—a reduction of about 22%. It was obvious that we had to strike some sort of an average, and it is always gratifying to obtain an increase in make over and above that obtained by the reduced coke rate.

* K. Neustaetter, *Proceedings of the Blast Furnace, Coke Oven and Raw Materials Conference, A.I.M.M.E.*, 1950, vol. 9, p. 303.

The figures Mr. Patchett quotes, of the increase in iron make and reduction in coke rate when using crushed and screened burdens as compared with unprepared burdens, are extremely interesting, particularly as they are comparable with the improvements we expect when our burdens are fully prepared and our sinter of the best quality. Our anticipated improvements are 2 cwt. off the coke rate and 20% increase in iron make.

It is understandable that Mr. Towndrow should show so much interest in the general operating conditions and should ask for more information. It was pointed out in the introduction to the paper that the emphasis was on methods of trial rather than on the figures obtained; however, Mr. Towndrow can be assured that we have not allowed ourselves to be led astray by the test results. During the 'good' weeks the scrap used was 4% of the burden—the usual amount at these furnaces. The flue dust showed a reduction of about 30%, *i.e.*, from a normal 3·4 cwt. to 2·4 cwt. per ton of iron. The slag volume was unchanged at about 24 cwt. per ton of iron. The furnaces were at all times given the amount of blast they would reasonably take, there being no question of controlling coke rates.

Mr. Towndrow correctly points out the outstanding effect of the use of Appleby-Frodingham coke. The realization of this effect led the Appleby-Frodingham Company to include new coke ovens in the extensions scheme. It was, however, unfortunate that the tests with good burdens were of such short duration, as it is felt that the effect of burden cleanliness is cumulative and would have become more apparent as the test continued.

The authors apologize for the error in the report on Experiment (5). The Thurcroft slacks were, of course, coked with a shorter carbonizing time. The final paragraph, referred to by Mr. Towndrow, was intended to convey that furnace performance with coke *A* was not as good as with normal Thurcroft coke, which always had a very pronounced effect on furnace performance.

Discussion on the Paper—
THE PRESENT POSITION OF THE CONVERTER PROCESS: ECONOMIC COMPARISON WITH OTHER STEELMAKING PROCESSES* *By B. Matuschka*

AUTHOR'S WRITTEN REPLY

(The Discussion on this paper and the author's verbal reply were published in the February, 1952, issue of the Journal. The written reply, printed below, was received too late for the inclusion in that issue).

Dr. **Matuschka** wrote in reply: I agree with Mr. Campbell that no generally applicable price calculation can be laid down, but I have taken care in my paper to use basic prices on which adjustments for locally applicable prices and conditions can easily be made. Where the price possibilities permit, more use should be made of the converter in steel production in order to master the great scrap scarcity in Europe.

The ECE Steel Committee (UNO) agree that the scrap shortage can be mastered only by raising the consumption of pig iron and by the increased use of direct iron-producing processes such as the Krupp-Renn, sponge iron, etc.

Fundamentally, my calculations agree with those of Dr. Suess. My figures should supply the basis for determining whether, in the future, the converter process can compete with the open-hearth process. In my comparison I have neglected amortization, which is limited by time, and which varies in different countries.

Amortization has, however, been estimated by Dr. Bading† as follows:

	Basic Converter	Open-Hearth (Pig-Ore Process)
Ingot Costs	£11 13*s.*	£11 11*s.*
Amortization	7*s.*	19*s.*
Total	£12 0*s.*	£12 10*s.*

Thus, including amortization, the costs of the basic Bessemer ingots are 95% of those of open-hearth ingots when the pig-ore process is used with the latter. The amortization must, however, be determined in each particular case.

As Mr. Kerlie says it is correct that with phosphoric pig irons a high lime rate of 150 kg./ton is required, but in the example quoted in Table 1*b* of my paper, the return from the slag is greater. Bading estimates a cost of 6*s.* 7*d.* for 147 kg. of lime, which is 2*s.* 7*d.* more than the figure I quote, but against this the gain through slag return is 16*s.* compared with 11*s.* 8*d.* in my calculation, *i.e.*, 4*s.* 4*d.* more. I assumed a pig-iron price of £10 per ton (120 DM). Bading gives 113 DM for basic pig iron. Open-hearth pig costs about 115 DM/ton in Germany. Scrap from the works is put in at a lower price than purchased scrap: Bading does this also.

Today scrap is charged in many blast-furnaces, but it should be used to cheapen the open-hearth steel. My calculated ingot costs agree well with those of Bading, although calculated before Bading's were published.

Dr. Suess has given the oxygen consumption for the oxygen-blowing process as 2120 cu. ft./ton. Dr. Durrer has also arrived at this figure, but used 99·5% of oxygen, which is much more expensive. The designs for the plant for the induction converter are mechanically and electrically ready for erection. The equipment is very simple and the cost of repairs is no higher than for other converters.

Data on the distribution of the German steel production were not available when this paper was prepared, but the figures for 1950 are compared with those for 1936–38 in Table *A*. There has been little change since 1950.

Mr. Colclough's assumption that the average scrap charge for European open-hearth furnaces is 75%, is not confirmed by the latest returns. The average figure for 1951, estimated in Geneva, is 66·2%, but owing to the scrap shortage it will scarcely exceed 60%, which is what it was before the war. Mr. Colclough may not have taken into account that today 14% scrap is charged into the blast-furnace and that, as shown in Table *A*, the production of electric steel has risen since the war from 4·5 to 7·3% of the total ingot production. (100% scrap is charged into the electric furnace.)

The assumption that an increase in steel production will lead in a few months to an increase in the amount of available scrap is based on a misconception. According to investigations by the Scrap Committee of ECE it is only the steelworks' circulating scrap, amounting to about 25% of ingot production (about half of the total scrap), which returns to the melting shop in a short

* J. Iron. Steel Inst., 1951, vol. 168, May, pp. 40–45.
† W. Bading, *Stahl u. Eisen*; 1951, vol. 71, pp. 373–388.

Table A—PERCENTAGE PRODUCTION OF PIG IRON, 1936–38 AND 1950

	Basic-Converter Process		Converter Process		Open-Hearth Process		Electric-Furnace Process	
	1936–38	1950	1936–38	1950	1936–38	1950	1936–38	1950
Austria	...	...	...	...	83	81·5	17	18·5
Belgium	85	87	1·5	0·7	12·5	10·2	1·0	2
France	64	63	0·7	0·9	30·3	29·9	5·0	6·1
Germany (West)	41	41·7	0·7	0·7	55	55	3·3	2·4
Luxembourg	98·5	97·3	...	...	0·3	...	1·2	2·7
Italy	...	...	1·0	...	71	60	28	40
Netherlands	...	...	...	...	...	98·8	...	1·2
Denmark	...	...	...	...	...	94	...	6
Norway	...		...	...	...	...	100	100
Saar District	76	74·0	0·3	0·1	22·7	23·6	1·0	2·2
Sweden	9	8·1	1·5	2·0	66	48·5	23·5	41·3
Switzerland							...	100
Turkey					...	100		
U.K.	4·0	5·2	2·0	1·5	92·0	87·7	2·0	4·5
European Average	39·0	35·4	1·0	0·9	55	55·1	4·5	7·3

time. Each year about 10% returns to the steelworks from the engineering and fabricating works. On the other hand, it takes 25–30 years for iron, after it has become unusable, to be available to the steelworks as a collected scrap. When there is a marked increase in steel production, as there has been in recent years, about one-third of the collected scrap arising from this greater production, is again available to the steelworks only after many years.

I do not quite understand Colclough's remarks on the open-hearth process : on p. 41 I have described the pig-ore process in the open-hearth furnace, and have stated that we still use this process in Austria. I have also mentioned the Talbot process. The application of a particular process of steel production varies, of course, with local conditions but the scrap scarcity is general throughout Europe. The scarcity will only be overcome when each country in which the proper technical conditions exist introduces scrap-saving steelmaking processes or applies them on a large scale.

The ECE Steel Committee is trying to solve such problems by united effort. The question of whether the broadening of steel production should be undertaken by putting up open-hearth furnaces or by producing more converter steel is being discussed. To what extent new methods and processes are to be used in different localities is left to individual investigations.

Regarding the manganese question, it is known that Dr. Röchling has made successful trials with his rotary refining vessel, in which the vanadium proportions of vanadium-bearing pig iron produced from Lorraine ores were transferred into the slag by blowing and were then recovered as vanadium alloys in the blast-furnace and alloy furnace. He also planned tests to obtain a manganese slag by blowing Donawitz pig iron in his refining vessel, but the tests could not be carried out in 1944 because of the war. As Works Director at Donawitz, in 1944 I planned to erect a similar refining vessel through which the pig iron was to run in a continuous process. The separation of manganese in a basic converter lining is only successfully achieved when the carbon content has been worked well down by blowing. This can also be seen from Colclough's data, obtained from tests in which a liquid converter iron with 1% of carbon and about 1% of manganese was produced. The carbon cannot, however, be blown from 4·0 to 1·0% in 4 min.; 8–9 min. are required. The time of 4–5 min. would therefore appear to be a mistake.

Trials which I made in 1947 with Styrian pig iron in an acid-lined Bessemer converter of 2 tons capacity at the Linz foundry showed that, under acid conditions, the manganese oxidized before the carbon. Although the manganese in the pig iron had dropped from 3·8 to 1·0% after 4 min., the carbon had only dropped from 4·0 to 3·8–3·9%. It was only after 8 min., when the Mn had dropped below 0·50%, that the carbon began to burn vigorously. For these reasons and because, when using an acid lining, the phosphorus in the manganese-rich slag, which is forming, is not taken up, I maintain that to blow a high-manganese pig iron in an acid converter in order to expel the manganese into a manganese-rich slag is an advantageous and proper method of producing good-quality ferromanganese. The trials which Mr. Colclough quoted are unknown to me.

The data in the heat table on p. 45 are quite correct. After blowing, the composition of the high-manganese iron is 3·8–4·0% C; 0·8–1·2% Mn; 0·1% P. After drawing off the high-manganese slag, the iron was cooled to 1300° C. The figures on p. 45 show that this iron could not be raised to the required pouring temperature by blowing either with air or with oxygen. This process therefore requires additional heating by induction.

CORRIGENDUM

THE FUNCTION OF ENERGIZERS IN PACK CARBURIZING—A CORRECTION

By A. Hultgren

In a recent paper[1] on pack carburizing, some experiments were also reported (Table IX) in which a gas mixture (50% N_2 + 50% CO_2) was passed through a heated vertical tube containing a 10-cm. layer of 1–3 mm. charcoal, with or without admixed energizer, the exit gas being analysed. Some of the results obtained are as follows :

Expt. No.	Addition	Gas Flow-Rate,* litres/min.	CO_2 in Exit Gas, %
2	None	0·5	19–20
7	0·8 g. NaOH	0·5	0
8	1·1 g. KOH	0·5	0
9	1·35 g. K_2CO_3	0·5	0·1
13	3 g. BaO	0·5	5·8
19	3 g. BaO	0·06	0·2

* At 925° C.

Manuscript received 9th October, 1951.

Table A

RESULTS OF ADDITIONAL EXPERIMENTS

Expt. No.	Carbon	Addition	Asbestos	Silica Gel	Temp., °C.	Inlet gas		Gas Flow-Rate, l./min.	CO_2 in Exit Gas, %
						N_2, %	CO_2, %		
21	40 g. graphite, 1–3 mm.	4·4 g. KOH	No	No	925	52·6	47·4	0·006–0·5	0·4–0·6
22	10 g. charcoal, 1–3 mm.	3 g. BaO	Yes	No	925	50·5	49·5	0·006	1·0
23	40 g. graphite, 1–3 mm.	12 g. BaO	Yes	No	925	50·5	49·5	0·02	2·7
								0·006	1·5
								0·002	1·6
24	10 g. charcoal, 1–3 mm.	0·8 g. KOH	Yes	Yes	925	50·5	49·5	0·006–0·5	0·8–1·0
25	40 g. graphite, 1–3 mm.	0·8 g. KOH	Yes	Yes	925	51	49	0·02–0·5	0·8–1·0

Since, under the conditions of the experiments, equilibrium with graphite is represented by a calculated value of 0·81% of CO_2, the low values obtained for CO_2 were surprising, and it was concluded that the CO_2 content was governed by a reaction in which the energizer took part and with a low equilibrium value for CO_2, and not by the carbon reaction. This condition was thought to apply also to the stationary atmosphere in a pack carburizing box.

However, it became apparent that the results might to some extent have been affected by experimental errors. Chipman[2] suggested that a loss of CO_2 in the exit gas might occur as a result of a reaction with alkaline matter picked up by the gas in passing through charcoal mixed with NaOH, KOH, or K_2CO_3. The reaction would possibly be assisted by cooling to lower temperatures.

It was also suspected that the exit gas was mixed with air. The gas samples for analysis were collected in 130-ml. vessels, with 4·5-mm. dia. outlets, originally filled with air. At a flow rate of 0·5 litres/min., the period of 10 min. used for flushing the vessel before analysing was undoubtedly sufficient, but at the slow rate of 0·06 litres/min. used in the BaO experiment it seemed probable that, although 30 min. were allowed, the gas collected was mixed with air.

In an attempt to avoid such errors, and to throw further light on the effect of certain energizers, a new series of experiments has been carried out as follows:

(i) Experiment 21 was carried out as in the previous investigation,[1] but using a similar volume of pure graphite (99·99%) instead of charcoal

(ii) In the following experiments using charcoal or graphite, a 5-mm. layer of asbestos wool was placed beneath the carbonaceous charge, next to the supporting diaphragm

(iii) In experiments 24 and 25, with KOH added, (*a*) a 30-mm. layer of silica gel, 1–3 mm. particles, was placed in the high-temperature region immediately below the exit diaphragm, with the object of removing any alkaline vapour that might have been picked up by the gas in the reaction chamber; and (*b*) during the heating-up period the nitrogen stream was directed upwards, so as not to waste silica. The reaction gas was then led in the opposite direction

(iv) The gas collecting vessels were filled with 10% NaCl solution.

The results are given in Table *A*.

The only CO_2 value obtained (in experiment 21) that is less than the equilibrium value with graphite, *i.e.*, 0·81%, indicates a probable loss by reaction with alkaline matter after the gas left the reaction chamber. Within the accuracy of the experiments, however, the equilibrium value of CO_2 for the graphite reaction was reached, in experiments 22 (charcoal + BaO), 24 (charcoal + KOH), and 25 (graphite + KOH).

In experiments 24 and 25, the silica gel appears to have effectively removed the alkaline vapour from the exit gas. Whereas, under the present conditions of gas flow, KOH appears to have been an efficient catalyser for establishing the graphite equilibrium for both charcoal and graphite, BaO was efficient for charcoal but not for graphite (experiment 23). The result of experiment 22 indicates that in the earlier BaO experiment (No. 19) the low CO_2 value of 0·2% was due to a sampling error.

It is believed that the accuracy of the new experiments is sufficient to prove that the earlier gas-flow experiments, using NaOH, KOH, or K_2CO_3 as an energizer, and the slow flow-rate experiments with BaO added,[1] were in error, for one or other of the two reasons indicated. That a difference in activity between charcoal and graphite is not discernible in the results of experiments 24 and 25 may be due to insufficient accuracy, and such a difference is perhaps visible in the results of experiments 22 and 23.

Of the earlier gas-flow experiments, at a rate of 0·5 litres/min., those with unmixed charcoal and with charcoal mixed with MgO, CaO, SrO, or BaO are believed to have given correct results.

In conclusion, the essential function of energizers in pack carburizing is probably to catalyse the reaction by which CO_2 formed at the steel surface is converted to CO to the extent demanded by the carbon equilibrium at the carburizing temperature.

The present gas-flow experiments, like the earlier ones, were carried out by Mr. K. Kuo, to whom the author is indebted for painstaking work and valuable suggestions. Dr. Chipman's suggestion is greatly appreciated. The pure graphite was supplied by Dr. U. Lamm, The Asea Co., Ludvika, Sweden.

REFERENCES

1. A. HULTGREN: *J. Iron Steel Inst.*, 1951, vol. 168, pp. 245–259.
2. J. CHIPMAN: Private communication.

B. I. S. R. A.

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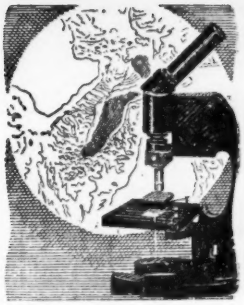

Projecting and Depressed Crazing of Ingot Moulds

A MICROSCOPICAL EXAMINATION AT THE INNER SURFACES OF 10-TON MOULDS

By R. E. Lismer, A.Met., A.I.M., and F. B. Pickering, A.Met., L.I.M.

SYNOPSIS

Two 10-ton ingot moulds, representative of the projecting and depressed types of crazing on the inner surfaces, which were associated with short and long tap-to-strip times respectively, have been examined. It is concluded that projecting crazing results from growth at the inner surface and from the development of an open network of deep cracks, whereas depressed crazing results from severe oxidation at the inner surface, growth being of subsidiary importance.

Introduction

During an investigation by Jackson[1] on the comparison of design and conditions of service of 10-ton ingot moulds, it was observed that the 'crazed' appearance of the inner mould surfaces depended upon the length of time that the hot ingots were held in the moulds. It was shown that a short tap-to-strip interval was associated with projecting crazing (Fig. 2*a*) and a considerably longer mould life, compared with a long tap-to-strip interval associated with depressed crazing (Fig. 2*b*).

Samples for examination, about 9 in. square, were taken through the wall thickness half-way up one of the walls of two moulds, representative of the projecting and depressed types of crazing.

Details of the moulds, given by Jackson, are recorded in Table I.

The analyses of the two moulds and the mode of failure were similar, although there was a difference in mould life.

MACROSCOPICAL EXAMINATION

Sulphur prints obtained on sections across the wall thickness were similar in appearance, and showed a pronounced dendritic pattern with marked segregation towards the outer and inner walls. The final wall thicknesses of moulds 36 and 61 were 7 and 6½ in. respectively.

Mould 36

The projecting type of crazing appeared to be a growth of the inner surface, with the development of an open network of cracks, the latter penetrating up to a depth of 1½ in. (Fig. 3*a*). The appearance of the crazing was reminiscent of the first stage in the growth of chrome-magnesite refractories.

Mould 61

The depressed type of crazing appeared to be the result of severe oxidation of the inner surface. The network structure of the cracks was finer than that of mould 36, and the cracks, which penetrated only to a depth of about ¼ in., were more in the nature of oxide inroads (Fig. 3*b*).

MICROSCOPICAL EXAMINATION

A thin slice extending from the outer to the inner surface was cut from each mould sample, and microscopical examination was carried out at positions up to 2 in. from the outer and inner surfaces, and also at the middle of the wall thickness.

Mould 36

The general distribution of the various structural features of mould 36 is shown in Fig. 1. The changes of the various micro-constituents from the outer to

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Table I

DETAILS OF MOULDS

Mould No.	Maker's Analysis, %					Life, number of casts	Mode of Failure	Type of Crazing
	Total Carbon	Si	Mn	S	P			
36	3·63	1·68	0·89	0·076	0·080	208	Horizontal crack on broad side	Projecting
61	3·63	1·68	0·56	0·064	0·070	115	Horizontal crack on broad side	Depressed

the inner surface, as the result of oxidation and temperature gradient effects, are discussed below.

Carbide—Adjacent to the outer surface and to a depth of about ¼ in., the matrix was almost completely decarburized, abruptly merging into a coarsely lamellar pearlite, which had been little or not at all affected by the temperature attained by the outer surface of the mould (Fig. 4*a*). With increase of temperature towards the inner surface, the first change in the appearance of the carbide consisted of spheroidization accompanied by a decrease in the amount of carbide present (Fig. 4*b*) until the structure became almost entirely ferritic. This reduction was probably caused by sub-critical graphitization,[2] yet no secondary graphite was visible on the original graphite flakes.

Near the middle of the wall thickness, the temperature attained in service appeared to be just above the Ac_1 point and small amounts of lamellar pearlite areas appeared in a grain-boundary formation (Fig. 4*c*). Nearer the inner surface, as the temperature further increased, the amount of pearlite gradually increased (Fig. 4*d*) until, at a position corresponding to a temperature just above the Ac_3 point, a fully pearlitic structure was obtained (Fig. 4*e*). From this position and towards the inner surface, oxidation around the graphite flakes had taken place, and at the periphery of the oxygenated areas, the carbide had coalesced (Fig. 4*f*) as an almost continuous layer, together with additional carbide particles present within the oxide zones. In places, the pearlite lamellae of the matrix structure were continuous with the coalesced carbide layer. Near the inner surface, the carbide had coalesced into larger particles interspersed between the graphite flakes and the oxidation products (Fig. 4*g*). Approaching the inner surface, the amount of carbide gradually decreased to zero.

The relatively low temperature reached adjacent to the outside surface of the mould had apparently been insufficient to materially affect the pearlite of the original as-cast structure. Where the temperature approached the critical range for the matrix, the structure became open, sub-critical graphitization and spheroidization having occurred, although in areas surrounding the phosphide constituents the carbide retained its dense appearance. Phosphorus produces a decreased solubility of carbon in ferrite,[3] and thus sub-critical graphitization would be prevented by restriction of the rate of solution of carbide.

Where the temperature attained was within the critical range, transformation to austenite enabled the carbide to be retained in solution, and on cooling after each casting the lamellar pearlite structure was re-formed. With increase in temperature, more austenite was formed, and an increased amount of carbide dissolved. Thus graphitization was apparently prevented.

As discussed by Benedicks and Löfquist,[4] the differential changes in volume between graphite and

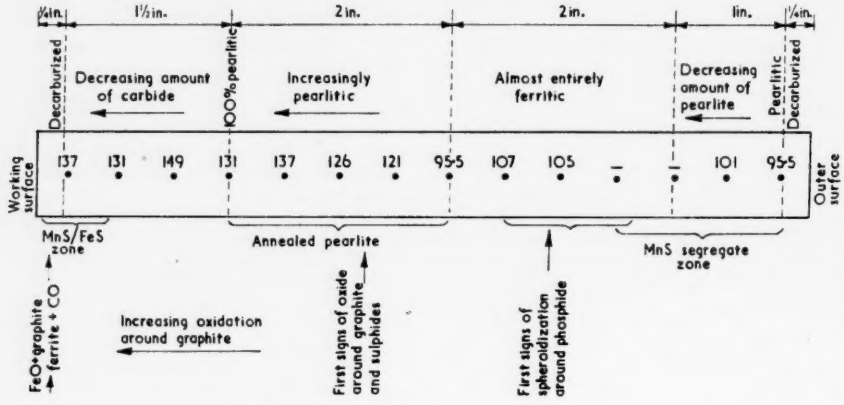

Fig. 1—Diagrammatic representation of changes occurring across the wall of mould 36 (the figures are the Brinell hardness determinations)

Fig. 2—Typical 'crazed' appearance of inner mould surfaces: (a) Projecting crazing; (b) depressed crazing

Fig. 3—Macro-etches obtained on sections across wall thickness: (a) Mould 36, showing cracking and oxide penetration at the inner surface; (b) mould 61, showing oxidation at the inner surface

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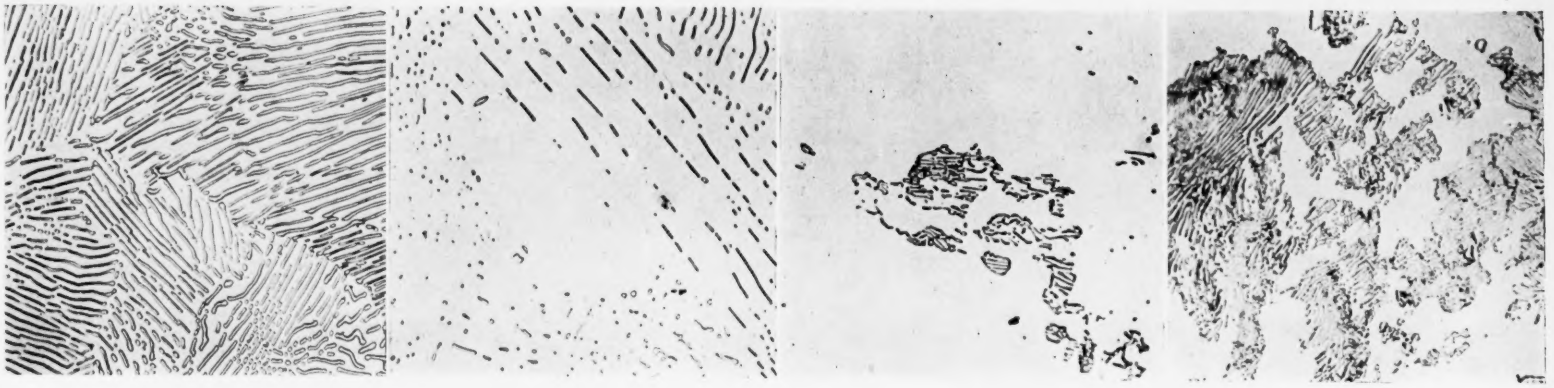


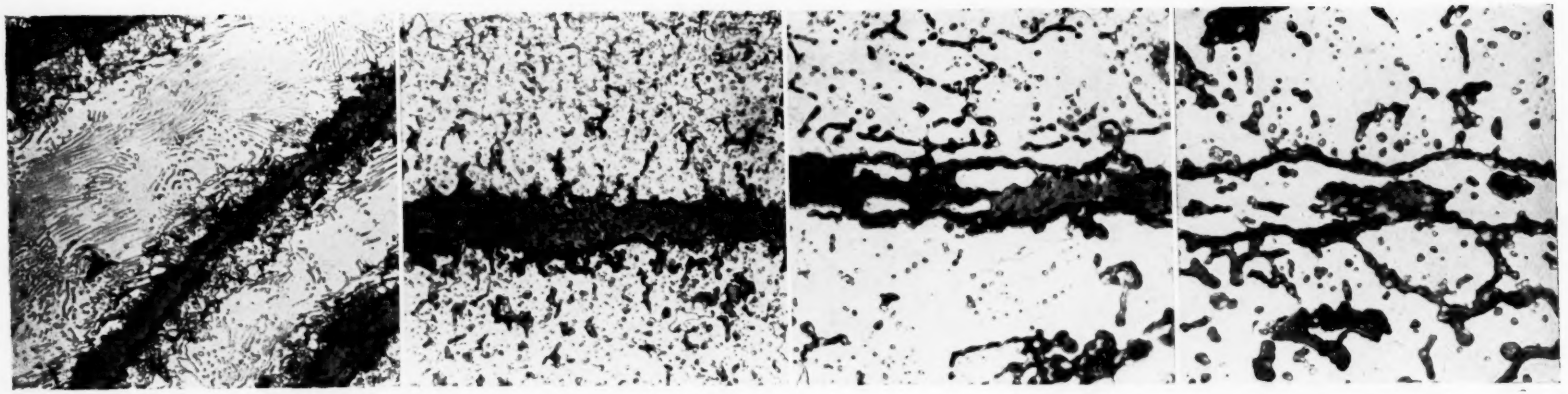
(a) (b) (c) (d)

(e) (f) (g)

(*a*) Unaffected pearlite matrix
(*b*) Sub-critical graphitization zone
(*c*) Grain boundary pearlite
(*d*) Increased amount of pearlite
(*e*) Completely pearlitic
(*f*) Coalesced carbide in presence of oxide
(*g*) Larger particles of coalesced carbide in presence of oxide

Fig. 4—Variation in carbide across wall thickness (mould 36) × 750

Fig. 4—Variation in carbide across wall thickness (mould 36) × 750



(a) (b) (c) (d)

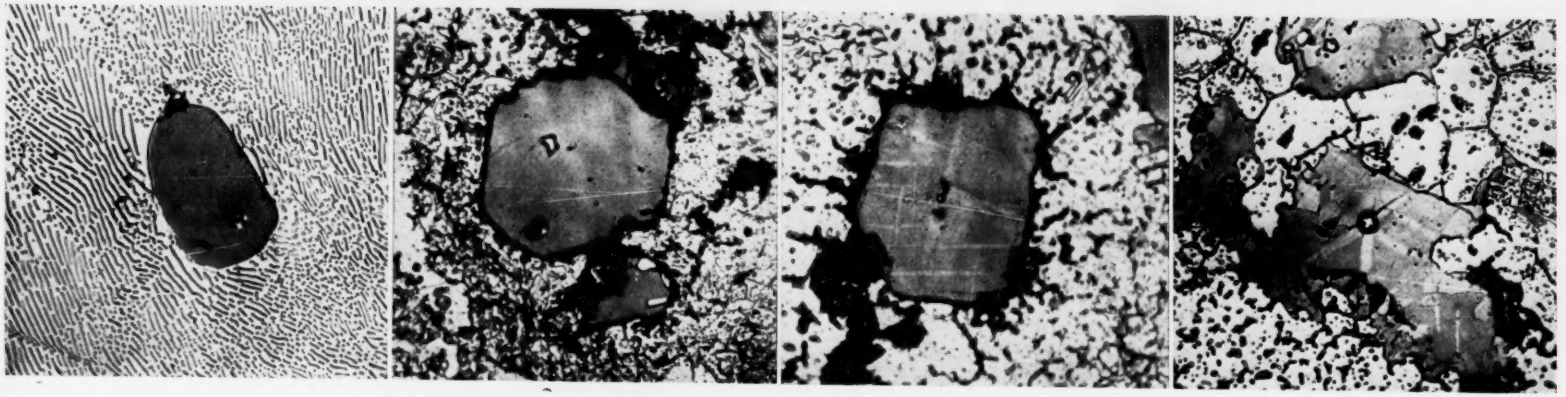
(*a*) Commencement of oxidation

(*b*) Commencement of oxide layer formation

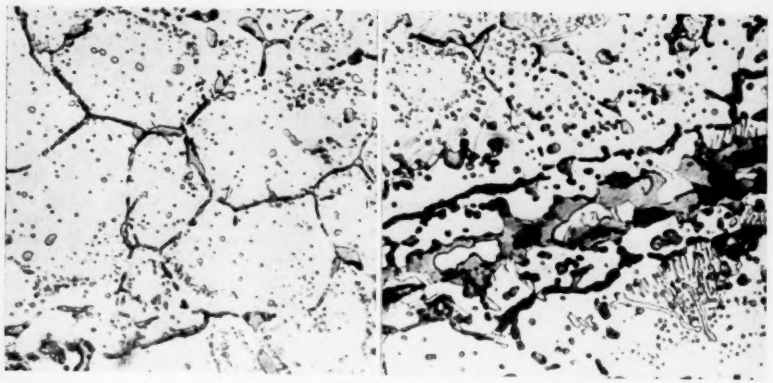
(*c*) Commencement of graphite-oxide reaction

(*d*) Completion of graphite-oxide reaction

Fig. 5—Variation in appearance of graphite flakes across wall thickness (mould 36) × 750



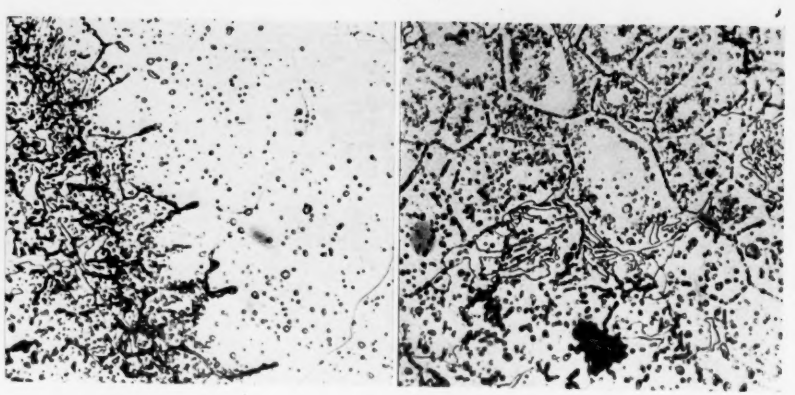
(a) (b) (c) (d)



(e) (f)

(*a*) Original MnS
(*b*) MnS surrounded by oxide
(*c*) Formation of FeS within MnS
(*d*) Gradual dispersion of FeS to grain boundaries
(*e*) FeS and oxide in grain boundaries
(*f*) FeS and oxide in position of the original graphite flake

Fig. 6—Segregation of MnS inclusion across wall thickness (mould 36)
× 750



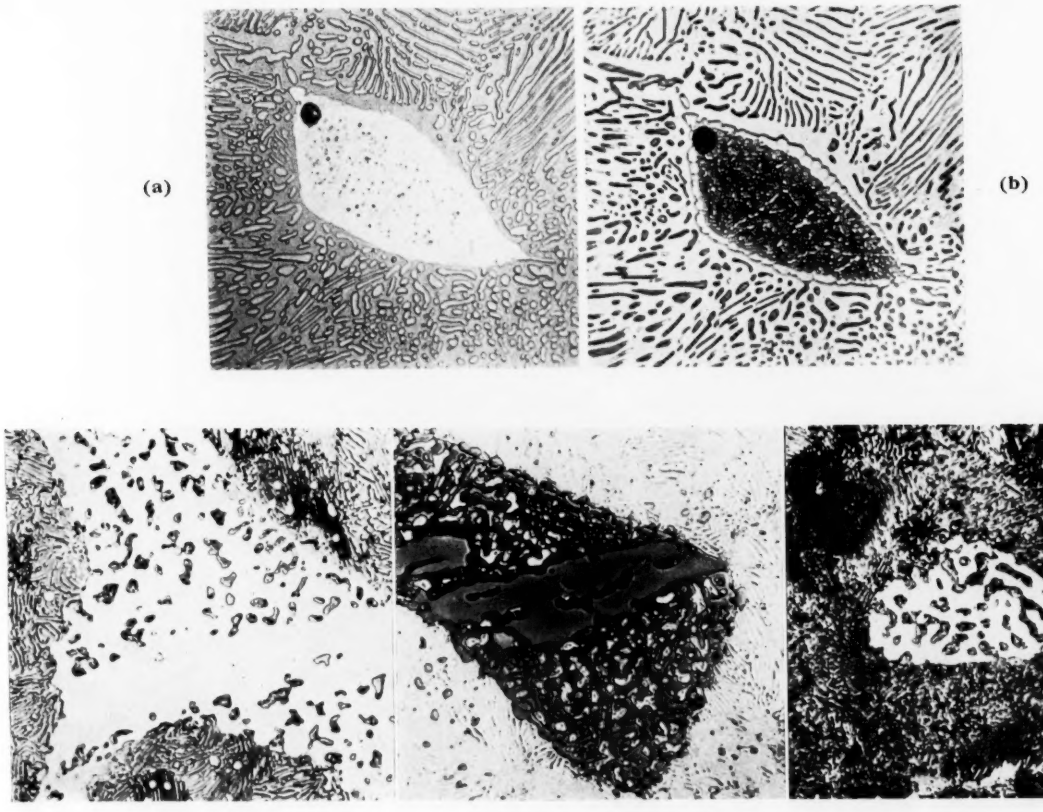
(a) (b)

(*a*) Intergranular oxide penetration
(*b*) Further oxide penetration

Fig. 7—Oxidation of iron across wall thickness (mould 36)
× 750

× 750

× 750



(a) (b) (c) (d) (e)

(*a*) Etched in picral

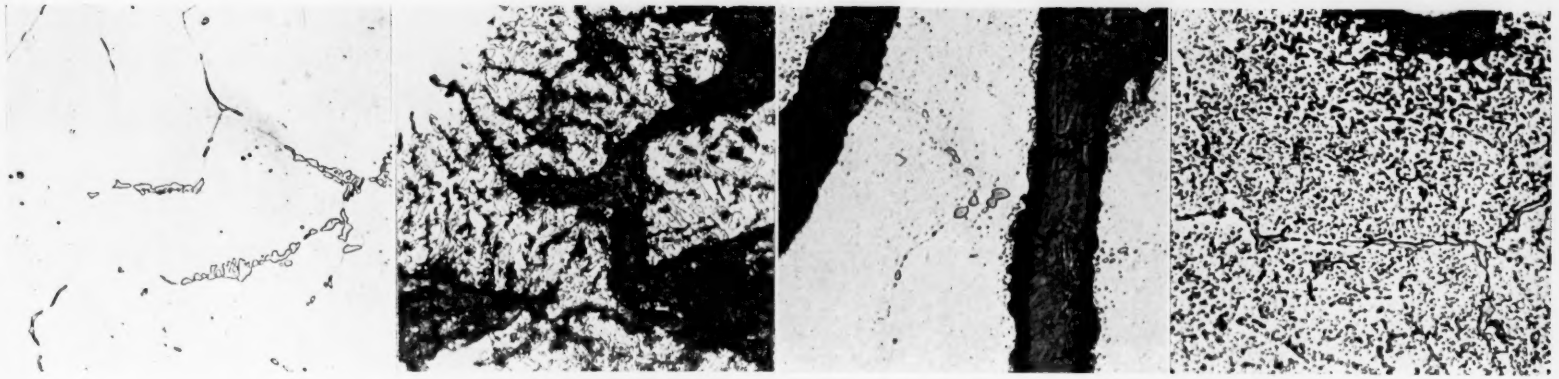
(*b*) Etched in picral, then in chromic acid followed by Murikami's reagent

(*c*) Pearlitic areas and sheroidized carbide in surrounding matrix

(*d*) Ternary eutectic and cementite crystals, etched in chromic acid followed by Murikami's reagent

(*e*) Binary phosphide eutectic adjacent to working surface

Fig. 8—Appearance of typical phosphide eutectic area (mould 36) × 750



(a) (b) (c) (d)

(*a*) Grain boundary carbide
(*b*) Further oxidation and carbide formation
(*c*) Compact oxide around graphite
(*d*) FeS in ferrite grain boundaries

Fig. 9—Matrix structure of wall of mould 61 × **750**

the metal, caused by different thermal expansions and volume changes at the critical points on heating and cooling through the critical range, would account for the development of small fissures at the graphite/metal interface. These would allow gases to penetrate into the body of the mould wall and to oxidize the metal. This phenomenon probably explains why no oxidation was observed until the temperature was known to be above the Ac_1 point, as shown by the reappearance of lamellar pearlite. The initial oxidation consisted of the formation of a feathery growth of metallic oxide around the graphite flakes without much apparent oxidation of the graphite itself. The presence of carbide particles within the oxidized zones may be caused by a lowering of the solubility of carbon in austenite, excess carbide being rejected from solution and coalescing into a massive state. On cooling, further carbide is formed as pearlite, which may be nucleated by the pre-existing carbide particles. Some concentration of carbon in front of the growing oxide areas caused the formation of a coalesced layer of carbide at these positions.

The presence of oxidation products and carbide in juxtaposition can be partly explained from the curves of free energy of formation of oxides at various temperatures. These show that iron oxidizes more rapidly than carbon below 700–750° C., whereas manganese and silicon oxidize before carbon, below temperatures of about 1300° and 1550° C. respectively. However, microscopical observations showed that oxide growth had taken place while the iron was in the austenitic condition. Nearer the working face, where the temperature has exceeded 750° C., the graphite and carbide were oxidized in preference to the iron. Hence, the amount of both graphite and carbide decreased to zero adjacent to the working surface, leaving a structure of iron and oxide.

The presence of carbide and oxide together seems unusual, and the preferential oxidation of iron before that of carbon or carbide, below 700–750° C., had been previously noted by Desch[5] in Ovifak iron.

Graphite—The original appearance of the graphite flakes remained almost unchanged from the outer surface to the middle of the wall thickness, although some change must have occurred as a result of the sub-critical graphitization of the carbide. At the position across the wall thickness, where the service temperature exceeded the Ac_1 temperature, oxidation commenced as a feathery growth spreading out from around the graphite flakes (Fig. 5*a*). As the temperature attained increased towards the inner surface, the amount of oxide increased with coalescence and with the later formation of a compact oxide layer around the graphite flakes (Fig. 5*b*). At even higher temperatures, this oxide layer had reacted with the graphite to form ferrite at the graphite/oxide interface (Fig. 5*c*); this reaction proceeded until the graphite had been entirely consumed leaving a layer of excess oxide around ferrite areas occupying the original position of the graphite flakes (Fig. 5*d*).

The formation of secondary graphite by the breakdown of cementite below the critical range was not observable in the microstructure. The exact nature of the oxidation products formed at higher temperatures was not determinable, but was believed to consist of oxides of iron, manganese, and silicon, whereas, except for a position adjacent to the working surface, the graphite was apparently unaffected. The coalescence of the oxidation products and the formation of an oxide layer around the graphite flakes may be the effect of temperature and time. However, above about 800° C., carbon will oxidize in preference to iron and the mechanism, suggested by the appearance of the microstructure, was that the graphite deoxidized the iron oxide according to the reaction $FeO + C \rightarrow CO + Fe$.

The iron first formed at the graphite/oxide interface and progressively grew towards the centre of the flakes until the graphite had been entirely replaced by ferrite.

Sulphide—Initially, the sulphide existed in the form of rounded, idiomorphic crystals of MnS, sometimes containing particles of titanium nitride or iron (Fig. 6*a*). Segregations of these inclusions were evident at the interface between the thin outer ferritic zone and the pearlite matrix, and extended intermittently up to 2 in. from the outer surface.

Where the first observable oxide growth around the graphite flakes occurred, a layer of oxide had formed around the sulphide inclusions (Fig. 6*b*) and had penetrated into the surrounding metal. Near the inner surface of the mould, the yellow ferrous sulphide, FeS-rich phase, appeared as a Widmannstätten configuration across the MnS particles (Fig. 6*c*). When all the original MnS had been replaced, the FeS phase began to disperse into the grain boundaries of the metal (Figs. 6*d* and *e*) and to associate with the oxide at the grain boundaries or in the positions originally occupied by the graphite flakes (Fig. 6*f*).

The first change in the nature of the constituents associated with the sulphides was the formation of a layer of oxidation products around the MnS particles, probably the direct consequence of fissures caused by the differential expansion of the matrix in respect of the inclusion. This oxide was probably MnO, since Mn oxidizes more readily than Fe. Since the sulphide was a solid solution of MnS.FeS, the removal of Mn as MnO would tend to increase the FeS content, the composition moving into the range within the MnS–FeS system, which shows a marked fall in solubility of FeS in MnS with decrease of temperature. Repeated heating and cooling, and the progressive oxidation of the Mn, eventually converted the whole particle to FeS, which, with the oxides of iron, was dispersed into the grain boundaries, either by fusion or diffusion processes with the probable formation of the low-melting-point eutectics with FeS and the oxides of iron.

The alternative explanation of the changes entails the assumption that the oxide layer was FeO, and that the diffusion of Fe into the MnS particle enriched the MnS solid solution in FeS. The formation of the Widmannstätten pattern of the FeS solid solution within the MnS solid solution has been previously noted by Wohrman.[6]

Iron and Oxide—Excessive oxidation of the iron, except that near the graphite, was not observed until a position near the working surface was reached. The initial oxidation consisted of an intergranular

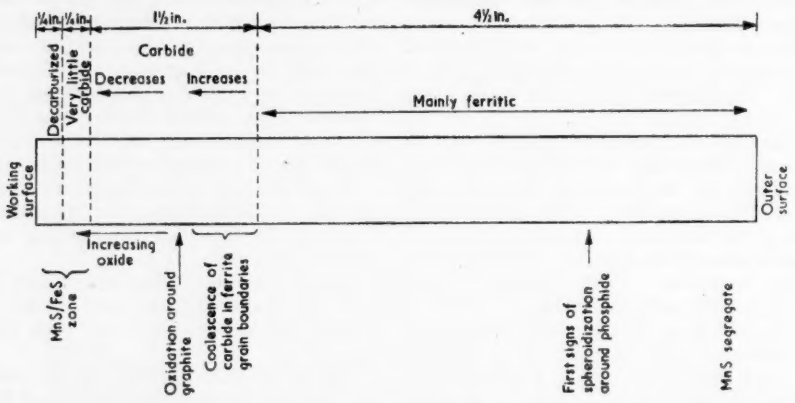


Fig. 10—Diagrammatic representation of changes occurring across the wall of mould 61 (Brinell hardness determinations < 95·5)

penetration of the austenite grains (Fig. 7*a*) from the oxide around the graphite flakes. This was followed by diffusion from the boundaries into either the austenite or ferrite grains (Fig. 7*b*). At higher service temperatures, the oxide had coalesced mainly at the grain boundaries and was associated with the grain-boundary migration of FeS. Such a highly oxidized matrix and the presence of brittle grain-boundary constituents would cause the metal to be very friable.

Phosphide—The phosphide areas near the outer surface of the mould appeared to be little or not at all affected by the lower temperature at this position. The appearance of a typical phosphide ternary eutectic area, etched in picral (Fig. 8*a*) and then in chromic acid followed by Murikami's reagent (Fig. 8*b*), had a true ternary eutectic structure of Fe, Fe_3C, and Fe_3P with an outer rim of carbide. The picral etch revealed only the ferrite (or transformed austenite) component. In other areas, large regular crystals or a mass of Fe_3C were evident, generally extending across the area.

The first visible change associated with the phosphide eutectic areas was a spheroidization of the surrounding pearlite, presumably because the phosphorus tends to stabilize the carbide. This spheroidizing and stabilizing effect was very marked and persisted with increase in service temperature to a value above the critical range. At this stage, the ferrite particles within the eutectic transformed to austenite, increased in size, and were able to take carbide into solid solution. On cooling, the austenite transformed to pearlite. Within the matrix surrounding the phosphide eutectic areas, complete solution of the carbide in the austenite was not achieved, the excess carbide being visible in the microstructure as spheroidized particles in a pearlite matrix (Fig. 8*c*). This figure also illustrates the definite pearlitic structure of the transformed austenite particles within the phosphide eutectic areas.

At higher service temperatures, the massive cementite crystals within the phosphide areas began to break down, probably because the austenite takes the carbides into solution (Fig. 8*d*). Near the working surface, the final change was a reversion of the ternary eutectic to the binary austenite (transformed to pearlite)–phosphide eutectic (Fig. 8*e*).

Occasionally, the phosphide areas, being the last to solidify, were associated with porosity and shrinkage cracks, and towards the inner surface these features assisted oxidation in and around the phosphide areas.

Mould 61

The general distribution of the various structural features is shown in Fig. 10. The changes were similar to those observed with mould 36, but the distance from the inner surface over which they were observed was considerably less. The matrix structure of mould 61 was mainly ferritic and the pearlite occurred predominantly at the grain boundaries (Fig. 9*a*) where the service temperature had just been above the Ac_1 point. This was the position at which oxidation commenced around the graphite flakes and the sulphide inclusions. However, since the matrix was mainly ferritic, within the areas of feathery oxide growth, the austenite had dissolved carbon, presumably from the graphite or penetrating CO gas, and on cooling had formed lamellar pearlite within the oxygenated areas (Fig. 9*b*). The carbon content of the austenite had apparently not attained saturation, and carbide layers around the areas of oxide growth were not evident. Adjacent to the inner surface, the graphite flakes became surrounded by a compact layer of oxide (Fig. 9*c*), but the zone showing the reaction between the FeO and graphite was not observed to the same extent as in mould 36, because this portion of the inner wall had eroded away during service.

Changes in the nature of the phosphide areas were similar to those in mould 36, although the final degree of oxidation of these areas was generally considerably greater. Changes of the MnS inclusions to FeS were similar, but, because they occurred over a much shorter distance, the development of Widmannstätten patterns was not so evident. The final stage of the sulphide change was the migration towards the

austenite grain boundaries (Fig. 9*d*). The changes of the ferrite and oxide constituents were similar to those of mould 36, although the oxide was generally more dispersed, and grain-boundary coalescence was less pronounced.

HARDNESS TESTS

The results of Brinell hardness determinations ($H_5/750$), carried out at $\frac{1}{2}$-in. intervals across the wall thickness of each mould are given in Figs. 1 and 10, respectively.

Mould 36, with short tap-to-strip intervals and the longer life, showed a pronounced increase of hardness towards the inner surface. An abrupt increase was apparent near the middle portion of the wall thickness, probably associated with a service temperature exceeding the lower critical point. Mould 61, with long tap-to-strip intervals, showed very low hardness values throughout. The influence of service conditions and life on the initial hardness of the moulds cannot be ascertained, although the differences in hardness values obtained on the two samples were possibly associated with different temperature gradients occurring throughout the wall thickness.

CONCLUSIONS

The examination carried out across the wall thickness from the outer to the inner surfaces of the two moulds has revealed that :

(1) Virtually the same structural changes of the micro-constituents occurred in both moulds.

(2) Projecting crazing was associated with growth at the inner surface and the development of an open network of deep cracks.

(3) Depressed crazing was associated with severe oxidation of the inner surface, and the development of a close network of shallow oxide inroads.

(4) Mould 36, which had a short tap-to-strip interval, had oxidized to a considerably greater depth from the inner surface than had mould 61, because of the nature of the oxidation rather than the longer life.

(5) Microstructural changes from the outer surface towards the inner surface of the moulds, were more gradual in mould 36 than in mould 61.

(6) Oxidation of the micro-constituents extended further from the inner surface in mould 36, but was more intense in mould 61.

(7) Hardness ($H_5/750$) from the outer to inner surface, showed a gradual increase from 95·5 to about 140 for mould 36, and a low value ($< 95{\cdot}5$) throughout mould 61.

DISCUSSION OF RESULTS

The mode of formation of the two types of crazing and the variations in the distribution of the microstructural features may be explained by a consideration of temperature and time effects during each individual life of the moulds, rather than by the difference between the total lives before failure in service. Mould 36, with a considerably longer life, showed less deterioration of the inner surface than mould 61, although the maximum temperature attained in both moulds was probably similar.

At the time of stripping mould 36, both moulds had the same temperature distribution, but thereafter mould 61 was at a higher temperature at all points throughout the wall thickness. The microstructural features and the extent of penetration of oxidation indicated, however, that the amount of wall thickness above the lower critical point (*i.e.*, partially austenitic) was greater in mould 36. No explanation can yet be offered for this apparent anomaly. The inner surface temperature at the time of stripping mould 36 was higher than that of mould 61 at its time of stripping, since the latter would have cooled during a longer period in the higher temperature ranges before stripping. This would also account for the greater penetration of oxidation in mould 36, although the quicker cooling through the critical range would suppress graphitization.

The higher degree of oxidation in mould 61 may, however, be associated with the longer period of time during which the ingot was in the mould and oxidizing gases were penetrating the gap between the mould and ingot surfaces. In addition, mould 61, possessing more total heat within the wall at the time of stripping, cooled more slowly than mould 36, with a result that the oxidation produced was more intense, but less deep. The quicker cooling rate of mould 36 also assisted the formation of deep cracks and thus the penetration of oxidizing gases was facilitated.

Acknowledgments

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References

1. A. Jackson : *J. Iron Steel Inst.*, 1951, vol. 167, pp. 289–301.
2. W. C. Heselwood and F. B. Pickering : *Ibid.*, vol. 168, pp. 277–286.
3. C. Benedicks and H. Löfquist : "Non-Metallic Inclusions in Steel," London, 1930 : Chapman and Hall.
4. C. Benedicks and H. Löfquist : *J. Iron Steel Inst.*, 1927, No. I, pp. 603–633.
5. C. H. Desch : *J. West Scotland Iron Steel Inst.*, 1925, vol. 32, pp. 40–44.
6. C. R. Wohrman : *Trans. Amer. Soc. Steel Treat.*, 1928, vol. 14, p. 81.

Absorptiometric Determination of Chromium in Iron and Steel

By the Methods of Analysis Committee

SYNOPSIS

Suitable conditions have been developed for the use of the diphenylcarbazide-chromate reaction for the determination of chromium in the presence of iron and most of the other elements occurring in alloy steels. Brief consideration is given to the limitations of other procedures.

THE present report describes an investigation into the development of a direct method for determining low chromium contents in iron and steel. Numerous volumetric methods for the determination of chromium have been published, but because of end-point errors they are unsuitable at very low chromium contents. These small percentages should be determined colorimetrically.[1, 2]

Of the colorimetric reagents for chromium examined, only diphenylcarbazide appeared worthy of serious consideration. The reaction between aniline hydrochloride and chromate[3] was examined, but was found to be so much inferior to diphenylcarbazide that further work on it was abandoned. The problem can be divided into two distinct parts: (i) method for oxidation of chromium; and (ii) definition of suitable conditions for the use of diphenylcarbazide as a colorimetric reagent. The latter is considered first.

The work was carried out by the Physico-Chemical Methods of Analysis Sub-Committee, constituted as follows:

H. G. Short, *Chairman* (N.P.L.); H. B. Anthony (Dorman, Long); D. Bell (Colvilles); H. C. Davis (R.A.E.); C. H. R. Gentry (Philips Electrical); S. Greenfield (G.K.N. Res. Labs.); H. Groom (Bragg Lab.); S. Harrison (Kaiser Ellison); G. Padget (United Steel Cos.); L. Russell (Babcock and Wilcox); K. Stanford (M.O.S., C.I.D.); R. J. Truman (Brown-Firth Res. Labs.)

COLORIMETRIC DETERMINATION OF CHROMIUM WITH DIPHENYLCARBAZIDE

The Reagent

Diphenylcarbazide is usually used as a solution in an organic solvent (acetone, alcohol, or acetic acid) and, when rather erratic results were obtained for chromium, it was thought that some reduction of chromate might have been brought about by the organic solvent before full development of the coloured complex with the reagent. A solution of the reagent in 3% aqueous phosphoric acid showed no improvement. Although a 0·04% solution could be prepared by dissolving the reagent in strong acid and diluting, solid tended to precipitate out on standing. In the present work a 0·25% solution in acetone has been used.

Supplies of diphenylcarbazide differed considerably in the amount of colour produced with a given amount of chromium, although any one supply gave consistent and reproducible results over a considerable period; nor could this difference in behaviour be correlated with the appearance of the sample. Some tests were made with recrystallized samples of diphenylcarbazide, but although the appearance of the product was improved there was no increase in the colour from a given amount of chromium, nor was the low figure given by a particular batch raised by recrystallization to the high figure originally given by another batch of reagent. That the effect was not caused only by the actual amount of diphenylcarbazide present in the test is shown by the following figures, which show that the colour given by a certain amount of chromium was only slightly increased by doubling the reagent strength, whereas another batch of reagent gave a much higher figure using the standard concentration.[4]

Effect of Reagent Concentration

Reagent	Spekker Difference (0·20% Cr sample)
A—5 ml. 0·25% solution	0·56
5 ml. 0·37% ,,	0·58
5 ml. 0·50% ,,	0·58
B—5 ml. 0·25% ,,	0·68

Reagent *B* had a melting point slightly lower than *A*

Any colorimetric determinations of chromium, using this reagent, had to be corrected by a calibration graph prepared from the same batch of reagent.

Interfering Elements

When an acetone solution of diphenylcarbazide is added to an acid chromate solution, the violet colour developed reaches a maximum in 5–10 min. and remains constant for some time. The only other elements giving coloured reaction products and likely to be present in steels are iron and vanadium, and in both cases the colour is extremely weak compared with that produced by oxidized chromium. The presence of vanadium, however, reduces the colour produced by a given amount of chromium, although the error only becomes significant in samples containing several per cent. vanadium and very little chromium, thus:

Added	Found
1% V, 0·2% Cr	0·190% Cr
1% V, 0·5% Cr	0·500% Cr
5% V, 0·2% Cr	0·165% Cr
5% V, 0·5% Cr	0·432% Cr

In neutral solution copper produces a colour similar to that given by chromium, but fortunately the colour is immediately destroyed by the addition of a small amount of mineral acid. With iron the solution is

Summary of paper MG/D/159/51 of the Physico-Chemical Methods of Analysis Sub-Committee of the Metallurgy (General) Division of the British Iron and Steel Research Association, received 10th September, 1951.

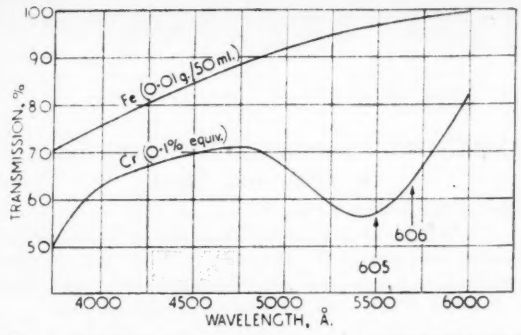

Fig. 1—Transmission curves of diphenylcarbazide complexes

initially almost colourless, but a brownish colour develops as the following figures, taken with a 2-cm. cell, 50-ml. volume, and a 606 filter, show :

Standing Time, min.	Spekker Difference Fe 0·02 g. Cr nil	Fe nil Cr 0·05 mg. (≡ 0·25%)
5	0·030	0·88
10	0·048	0·88
20	0·064	0·88
30	0·080	0·88

Whilst the chromium colour soon reaches a maximum and remains constant, iron produces an increasing background colour. From a consideration of the absorption values of the complexes (as shown in Fig. 1) it may be seen that a filter with maximum transmission at 5500 Å. (Ilford 605) is the most suitable for the measurement of chromium. The absorption of the solution caused by iron is, however, less with a 606 filter, with maximum transmission at 5700 Å. which is still near the peak of the absorption curve of the chromium complex. With this filter any variations in iron content will have a negligible effect on the calibration graph.

The addition of phosphoric acid before the addition of diphenylcarbazide considerably reduces the colours produced by iron and vanadium and has very little effect on the colour produced by chromium as the following figures show :

Phosphoric Acid Solution	Spekker Difference after 15 min. Standing Fe 0·02 g. Cr nil	Fe nil Cr 0·05 mg. (≡ 0·25%)	Fe nil V 1·0 mg. (≡ 5%)
Nil	0·05	0·73	0·030
30 ml. 2%	0·025	0·725	...
30 ml. 4%	0·015	0·725	0·020
30 ml. 8%	0·010	0·725	0·020

These figures were obtained with a 2-cm. cell, 50-ml. volume, and 606 filter.

A still greater amount of phosphoric acid causes a decrease in the chromium colour and in the final method an addition of 25 ml. of 4% acid has been used. Under these conditions the iron blank is considerably reduced and the chromium colour remains unaffected.

OXIDATION OF CHROMIUM TO DICHROMATE

Ammonium Persulphate as Oxidizing Agent

The first method to be investigated by the Sub-Committee was the widely used oxidation with ammonium persulphate, catalysed by the addition of silver nitrate. Sodium nitrite was used for reducing the permanganate formed, but, however carefully this was added, there was evidence that some reduction of chromate also occurred, and any excess caused serious error. The addition of urea has also been recommended for the reduction of traces of perchromates formed in the oxidation, but it was very difficult to obtain urea free from traces of chloride which cause troublesome turbidities with the silver.

Uncatalysed persulphate oxidation failed in the case of high manganese steels, where considerable precipitation of hydrated manganese dioxide occurred.

Potassium Bromate

Potassium bromate as oxidizing agent[5] has the advantage that manganese is not oxidized beyond the tetravalent state. In the original method excess bromate was destroyed by boiling with ammonium sulphate, the last traces being removed by further boiling with hydrochloric acid, and when this procedure was applied to samples of various steels the results obtained by the Sub-Committee were quite good, as the following figures show :

Operator	Chromium, % Sample 1	Sample 2	Sample 3
1	0·16	0·06	0·15
	0·16	0·065	0·145
	0·165	0·065	0·155
2	0·162	0·053	0·142
	0·161	0·053	0·136
	0·153	0·053	0·139
3	0·155	0·057	0·145
	0·153	0·057	0·145
	0·170	0·065	0·15
4	0·168	0·067	0·152
	0·172	0·070	0·150
	0·172	0·070	0·150
5	0·165	0·056	0·148
	0·165	0·057	0·148
	0·167	0·057	0·148

This method could not be recommended, however, because

(i) There was some evidence that, under the conditions used, slight reduction of chromate was occurring during the period of boiling with hydrochloric acid to remove the last traces of bromine, as follows :

Concentration of Hydrochloric Acid, % v/v	Time of Boiling, min.	Spekker Difference
Nil	5	0·323
0·5	2	0·293
0·5	7	0·262
0·5	15	0·183

Small amounts of bromine did not give any colour with the chromium reagent, nor did they appear to affect the colour produced by a given amount of chromium, but the background colour caused by iron was increased. Some tests were tried without hydrochloric acid, relying on ammonium sulphate to remove bromine, but the calibration curve and results were less reproducible than before.

(ii) Individual points on the calibration graph showed more scatter than is usual and an investigation of minor points in procedure gave no improvement in the definition.

In addition, the bromate method was comparatively tedious in operation and the examination was therefore suspended.

Perchloric Acid

Perchloric acid has previously been recommended for the determination of low chromium contents in iron and steel,[1, 6] but in these cases the intensity of the yellow chromate colour itself was measured. On examination it was found that perchloric acid did not interfere with the chromate-diphenylcarbazide reaction, and a number of samples were tested by a method involving perchloric acid oxidation followed by a colorimetric determination of chromium. No oxidation of manganese occurred.

The results were encouraging, but some members reported low figures and it was obvious that the conditions for the complete oxidation of chromium were very critical. After concentration of the solution to fumes of perchloric acid, the oxidation of the chromium occurred quite suddenly and could usually be determined visually from the colour change of the solution. Two or three minutes further boiling was then sufficient, but the solution had to be boiling and not merely simmering. Prolonged boiling gave low results for chromium. This critical procedure is described in greater detail as follows :

> Fuming of the perchloric acid commenced as a white cloud which persisted for several minutes within the covered beaker. The cloud suddenly cleared and was followed by almost invisible fuming in the beaker, with dense white fumes issuing from the lip. The solution was boiled for a further two minutes and no longer.

When the oxidized solution was cooled and diluted, some laboratories reported the presence of small amounts of reducing substances, leading to slightly low results for chromium. This effect was demonstrated by the decolorization of a small amount of permangate added to the solution immediately after dilution. This addition was therefore incorporated as a standard feature of the method. When the procedure was strictly followed it was found to give highly reproducible results for chromium.

The following percentages of chromium were obtained on six different circulated samples by the method of the Appendix, using the above procedure :

	Type of Sample					
Operator	0·8% Mn 1·0% Ni	1·5% Mn 0·3% W	1·5% Mn 0·3% Mo	0·75% Mn	Mild Steel	1% Ni
1	0·27	0·15	0·15	0·020	0·11	0·012
2	...	0·16	0·15	0·024	0·11	0·019
3	0·275	0·165	0·155	0·022	0·11	0·019
4	0·26	...	0·145	0·025	0·095	0·015
5	0·26	0·16	0·16	0·026	0·115	0·018
6	...	0·16	0·155	0·023	0·11	0·016
7	0·275	0·15	0·15	0·021	0·11	0·013
8	0·28	0·155	0·15	0·021	0·105	0·015
9	0·285	...	0·155	0·021	0·105	0·016
10	...	...	...	0·025	0·11	0·020
Average	0·27	0·155	0·15	0·023	0·11	0·0165

These figures are considered sufficiently satisfactory to recommend the procedure as a suitable method for low chromium contents ($< 0·3\%$) in iron and steel.

References

1. Brit. Iron Steel Res. Assoc., Methods of Analysis Committee : *Metallurgia*, 1948, vol. 37, pp. 211–216.
2. A. Portevin : *Compt. Rend.*, 1938, vol. 206, p. 518.
3. I. V. Tananaev and K. A. Matveeva : *Analyst*, 1948, vol. 73, pp. 173–174.
4. A. Bacon and H. C. Davis : *Journal of the Society of Chemical Industry* (*J. Appl. Chem.*), 1948, vol. 67, pp. 316–331.
5. M. Z. de Lippa : *Analyst*, 1946, vol. 71, pp. 34–37.
6. L. Singer and W. A. Chambers : *Indust. and Eng. Chem.* (Analytical Edition), 1944, vol. 16, pp. 507–509.

APPENDIX—Determination of Chromium in Iron and Steel

Special Apparatus Required

The Hilger photoelectric absorptiometer or other similar instrument is suitable. With the former instrument, a mercury vapour lamp, Ilford yellow 606 and H503 filters, and 4-cm. cells should be used.

Solutions Required

Nitric acid (2 : 3)
Potassium permanganate (0·5%)
Phosphoric acid (1 : 24)
Diphenylcarbazide (0·25% in acetone).

Procedure

Dissolve 0·25 g. of sample (Note 1) in 5 ml. of nitric acid (2 : 3), add 5 ml. of perchloric acid (sp. gr. 1·54) and evaporate to fumes. Maintain the solution at boiling point until the dense white fumes are driven out of the beaker and perchloric acid condenses at least one inch above the surface of the liquid. Cool, add one drop of potassium permanganate solution (0·5%) and 40 ml. of water, boil for 2 minutes and cool. Dilute to 250 ml. in a graduated flask, mix, and allow to stand for ten minutes.

Pipette 10 ml. into a 50-ml. graduated flask, add 25 ml. of phosphoric acid (1 : 24) (Note 2). Adjust the temperature of the solution to 20 ± 1° C. and add 5 ml. of diphenylcarbazide solution, dilute immediately to 50-ml., mix and allow to stand for 10 minutes. Measure the absorption of the solution using the conditions specified.

Calibration

Primary Chromium Solution—Dissolve 0·7071 g. of potassium dichromate in water and dilute to 1 l. 1 ml. of this solution contains 0·00025 g. of chromium.

Dilute Chromium Solution A—Dilute 100 ml. of the primary chromium solution to 1 l. 1 ml. of this solution contains 0·000025 g. of chromium.

Method—To several 0·25-g. samples of pure iron, add varying amounts, up to 25 ml., of solution *A* to cover the range 0–0·25% of chromium.

Treat each sample exactly as described in the ' Procedure ' and plot the absorption readings obtained against chromium percentage present to produce a calibration graph from which analysis results can be read off. Replicate results should lie within ± 0·01% of the true figure.

Notes

(1) For pig and cast iron, dissolve 1 g. of sample in 20 ml. of nitric acid (2 : 3), filter on a paper-pulp pad and wash with hot water. Cool and dilute the filtrate to 100 ml., pipette 25 ml. into a 250-ml. conical beaker, add 5 ml. of perchloric acid (sp. gr. 1·54) and continue as in the ' Procedure.'

(2) Large amounts of vanadium (several per cent.) cause slightly low results for chromium. The effect may be decreased by using 25 ml. of 1% phosphoric acid, but the colour should be read from a graph produced under similar conditions.

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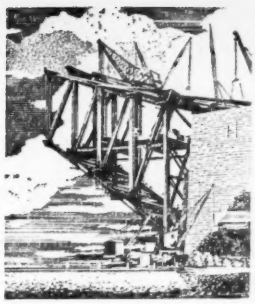

Supply and Use of Hydraulic Power at Appleby-Frodingham

By L. N. Lloyd, M.Inst.C.E.

SYNOPSIS

The Appleby-Frodingham Steelworks comprises two steel sections, one at the Appleby works, which includes a melting shop, a slab mill, and a reversing plate mill, the other at the Frodingham works, which is a similar plant operating a section mill. At the Appleby works there is a central pumping station with four 5-stage centrifugal pumps delivering water at 750–800 lb./sq. in. into a 10-in. dia. manifold. From this, an 8-in. dia. main carries water to the nearby melting shop at a central point where there is a 345-gal. accumulator. Another 8-in. dia. main takes water to the plate mill, and this has a similar accumulator about half-way down the mill and 774 ft. from the pumps. A smaller main serves the slab mill. The plate mill comprises two stands, roughing and finishing, for plates up to 12 ft. wide by 0·180 in.–2 in. thick and slabs up to 8 in., and two stands for plates up to 7 ft. wide by 0·120 in.–½ in. thick.

HYDRAULIC POWER SUPPLY

About 11 years ago the hydraulic system in the Appleby Steelworks was not satisfactory, owing to the difficult conditions then prevailing. At times the mill accumulator would fall on to its bottom blocks and the heavy demands of the shearing machinery would cause pressure drops of such magnitude that the rolling mill balance cylinders could not support the top roll assembly. An investigation was carried out, and modifications were made which resulted in a steady pressure, a floating accumulator at 750 lb./sq. in. in place of 730 lb./sq. in., and the load being carried by two pumps instead of three. This was a considerable saving, as the 'no-load' horsepower of one of these pumps is 300 h.p., and there are now two pumps working in their high-efficiency range.

The first step in the investigation was to ascertain the nature of the pressure fluctuations and how they could be mitigated. This led to tests of the pumps and to methods of improving their efficiency.

SUPPLY MAINS HEAD

Acceleration Head

In a large system of this sort, where the plate mill main is 1366 ft. long, considerable masses of water have to be accelerated. They are as follows :

Section of Main	Distance, ft.	Pipe Bore, in.	Weight of Water, tons
Pumps to mill accumulator	774	8	7·5
Accumulator to 2 in. × 12 ft. cross-cut shears	288	8	2·8
Cross-cut shears to two sketch shears	304	8 and 6	1·8
		Total	12·1

To ascertain the force required to accelerate or decelerate such large masses, pressure-gauge readings were taken at selected points in the main. Also, time/displacement graphs of a shear ram were made and the water demand rate was calculated. These results were then analysed by means of the following formula :

$$P = \frac{0{\cdot}0052\,QL}{AT},$$

where : P = acceleration head, lb./sq. in.
Q = change of flow, gal./min. (*i.e.*, initial flow subtracted from final flow)
L = length of pipe on centre-line, ft.
A = area of pipe, sq. in.
T = time, sec., for change of flow to take place.

This formula, expressed graphically in Fig. 1, is based on the assumption of a constant rate of acceleration ; its derivation is shown in Appendix I. The dotted line shows the method of using the graph. The radial lines to the right of the transfer line are based on pipe bore, and those on the left are based

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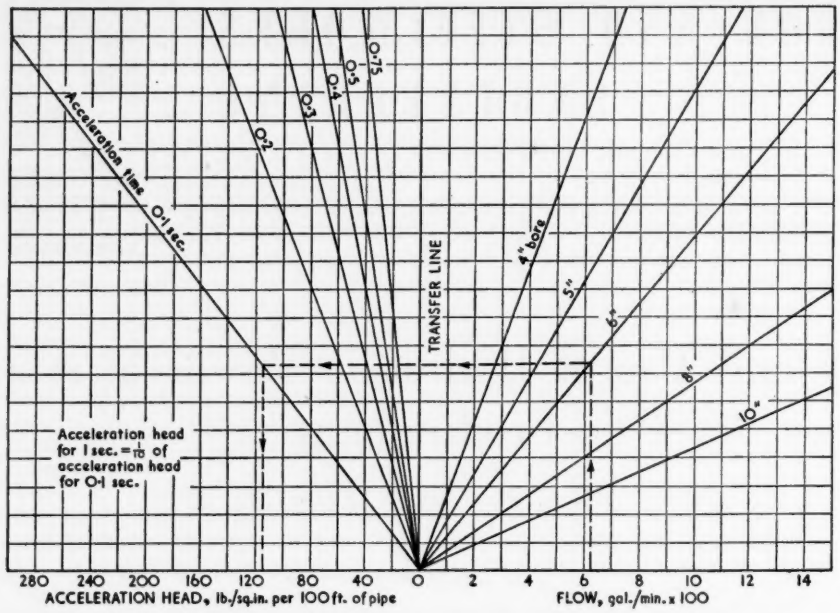


Fig. 1—Flow/acceleration-head graphs for water at 62·35 lb./cu. ft.

on acceleration time. Change of velocity could be substituted for Q if desired.

For any specific increase in flow, the acceleration head varies inversely as the area of the pipe ; also, the change of velocity is less in the larger-bore pipe. Thus, in problems where a small acceleration head loss is required, this can be achieved by accelerating a larger mass of water in a larger-bore pipe.

Friction Head

Acceleration head has a greater bearing on mill problems than friction head. Nevertheless, friction-head/loss must be also considered, and can be obtained from the graphs in Fig. 2, which are based on the well-known Reynolds Criterion formulae given in Appendix I. These graphs refer to turbulent flow in clean pipes of various bores. At high pressures the actual bore may be less than the nominal size, and a trial calculation should be made to see whether the difference is significant. Also, in a pipe system that has been in use for a number of years the surface may be roughened and the area reduced by corrosion, deposits from the water, or vegetable growths. Figure 3 shows the effect of corrosion on the friction factor in two 8-in. dia. pipes conveying petroleum spirit ; clearly, allowance should be made for corrosion effects when designing a hydraulic pipe system.

According to Fig. 2, higher velocities can be permitted in large pipes than in small ones. For example, a velocity of 20 ft./sec. in a 4-in. dia. pipe will cause a friction head of 14 lb./sq. in. per 100 ft., whereas in an 8-in. dia. pipe the loss is only 5 lb./sq. in. per 100 ft. In practice, the small pipes are generally used on short branch lines where a considerable pressure loss can often be tolerated. The designer must therefore consider the conditions in each section of the pipe system and fit pipes accordingly, and for this purpose the graphs in Figs. 1 and 2 will save much time.

Table I

PRESSURE CHANGES IN MAIN AND IN CROSS-CUT SHEARS

Accumulator : weight-loaded at 730 lb./sq. in. Pumps : 5-stage centrifugal.

Location of Pressure Gauge	Distance from Pumps, ft.	Normal Pressure, lb./sq. in.	Pressure Fluctuations during Cutting Stroke of Shears, lb./sq. in.		
			Start	Intermediate	Finish
8-in. dia. main at electric sub-station	489	762	674	...	840
8-in. dia. main at accumulator branch	774	765*	616	...	870
8-in. dia. main at branch to shears	1062	762	545	670	870
Power cylinder at shears cutting ¾-in. and 1-in. plate	1077	...	150	200/250	400

* The accumulator shows a higher pressure than the static load because it was up against the top stop with the relief valve open

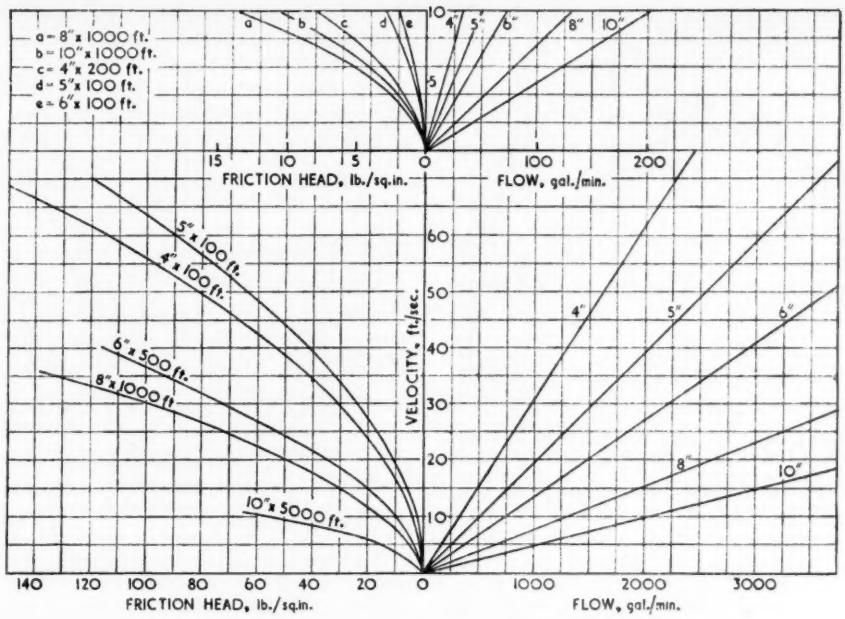


Fig. 2—Flow/velocity/friction-head graphs for water at 50° F.

Observed Acceleration Heads and Calculated Friction Losses

Table I is a summary of observed pressure changes at three points in the 8-in. dia. main and in the power cylinder of the 2 in. × 12 ft. cross-cut shears. These shears were the only load on that part of the system during the observations, and therefore pipe friction losses were low. The speed of the power cylinder rams of the shears was timed by a stop-watch, and the velocity is shown in Fig. 4, curve *a*.

In view of the head required to accelerate the 10·3 tons of water in the 8-in. dia. main from the pumps to the shears, it is evident that to start the shearing machine it must be able to begin moving under a lower pressure than the static pressure in the main, and that the lower this pressure, the greater will be the rate of acceleration of the water in the main. This fall in pressure at starting reduces the effect of the apron balance cylinder. The fall of the apron is thus aided by its weight, and this, in turn, increases the pressure drop in the main and aids acceleration.

Multiplying the ram displacement of the shears by the constant velocity given in Fig. 4, and applying the pressure difference in lines 2 and 3, column 4 of Table I, to the acceleration head formula, the following information is obtained :

Ram displacement	760 gal./min.
Pressure drop in main between accumulator and shear branch ...	71 lb./sq. in.
Weight of water in main between accumulator and shear branch ...	4·5 tons
Acceleration time	0·32 sec.
Pressure available at accumulator (*i.e.*, static pressure − line pressure)	114 lb./sq. in.

The acceleration time agrees well with the velocity

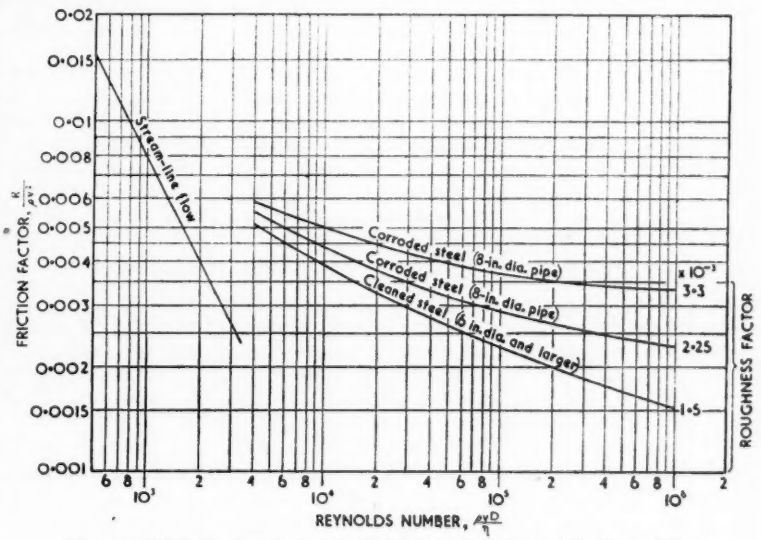


Fig. 3—Effect of corrosion on the friction factor in old pipe lines. (By courtesy of Anglo-Iranian Oil Company)

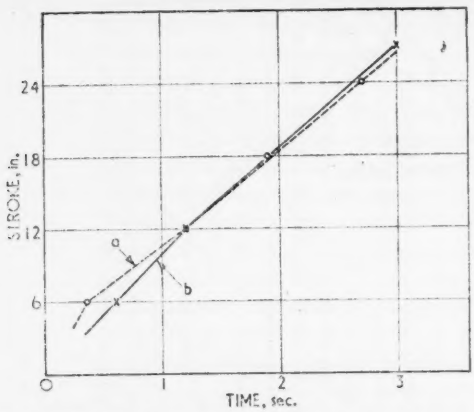


Fig. 4—Velocity graphs for ram of 2 in. × 12 ft. cross-cut shears. Curve *a* is for the original shears (constant velocity 0·666 ft./sec., 6–24 in. range); curve *b* is after alterations to piping (velocities 0·83 ft./sec. in 0–12 in. range, 0·77 ft./sec. in 12–24 in. range)

graph in Fig. 4. There is a considerable pressure difference at the accumulator between its static loading and the line pressure, and this is utilized in meeting the following requirements :

(i) Friction of ram stuffing box
(ii) Accelerating water in cylinder and branch to main
(iii) Water friction at cylinder exit and in the branch
(iv) Force to accelerate accumulator ballast weighing 173 tons.

In regard to item (iv), the accelerating force is provided by the difference in hydraulic pressure between the static loading of the accumulator and the observed line pressure at the branch connection to the main after deducting losses (i) to (iii).

Recent observations enable a rough estimate to be made of the distribution of supply between the pumps and the accumulator during the acceleration period. Observations have also been made of accumulator discharge during steady flow conditions (*see* p. 276).

In addition to the pumps now maintaining a steady pressure, the power and balance cylinders of the cross-cut shears have been redesigned and the hydraulic properties of the branch piping improved. The new velocity graph of the power rams is shown in Fig. 4, curve *b*, and pressure observations are given in Table II. During the test, the cross-cut shears was the only load applied, and between cuts the accumulator was at the top of its stroke with the relief valve open. Two 5-stage centrifugal pumps were used, and their discharge characteristics are given in Fig. 5.

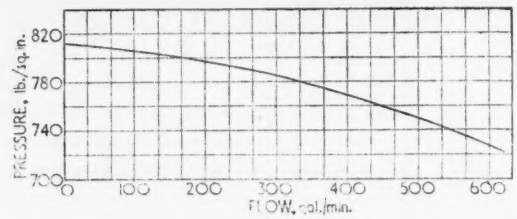


Fig. 5—Discharge characteristics of 5-stage centrifugal pumps

From Fig. 4, curve *b*, velocity of ram at end of acceleration period	0·83 ft./sec.
Net demand of shear power cylinders per 12-in. stroke after deducting returns of balance cylinders ...	21·2 gal.
Rate demand of shear power cylinders per 12-in. stroke at 0·83 ft./sec. ram speed	1060 gal./min.

Applying acceleration and friction-head formulae, and allowing for accumulator losses and for the fall in pump head that accompanies increase in flow, the following approximate results are obtained :

Accelerating time	0·212 sec.
Discharge from pumps at end of acceleration period	280 gal./min.
Discharge from accumulator at end of acceleration period	770 gal./min.

In regard to the fall in pressure, at the accumulator branch, of 98 lb./sq. in. below the static loading of the accumulators (*see* Table II), 59 lb./sq. in. was utilized to accelerate the accumulator ballast and the remainder to overcome gland friction and hydraulic resistances.

The high rate of flow from the accumulator is only transitory ; at the end of the acceleration period the mains pressure quickly rises and the discharge falls to an average of 400–500 gal./min.

RETURN MAINS

The return main at the Appleby mill starts with an 8-in. bore for the first 790 ft. and finishes at 10 in. for the last 406 ft. The acceleration problem was overcome in a simple way. Each of the large hydraulic shears discharges into surge tanks placed in the roof valley gutter, and these tanks discharge by gravity into the return main. This reduces the consumption of high-pressure water, since smaller balance

Table II

PRESSURE CHANGES IN MAIN AND IN CROSS-CUT SHEARS AFTER ALTERATIONS TO PIPING

Accumulator: weight-loaded at, 750 lb./sq. in. ; mass of ballast, 178 tons ; ram dia., 26 in. ; ram stroke, 15 ft. ; capacity, 345 gal.

Location of Pressure Gauge	Distance from Pumps, ft.	Normal Pressure, lb./sq. in.	Pressure Fluctuations during Cutting Stroke of Shears, lb./sq. in.	
			Start	Finish
8-in. dia. main at accumulator branch	774	775	652	850
8-in. dia. main at 2 in. × 12 ft. shears	1062	775	500	900/1000
Shears cutting 72 in. × ½ in. plate	...	...	150	250

cylinders can be employed, and speeds up the return stroke of the shears. Although all other users in the mill are coupled directly to the return main, those in the vicinity of the shears gain an advantage from the surge tanks, since the head in the main cannot be greater than that of the tank; moreover, the tank can take reflux water from the main. Between the mill and the pump house, other users have only 300–400 ft. of 10-in. dia. main to the return tanks, so that the acceleration head will be small.

The mean static head of the surge tank at the 2 in. × 12 ft. cross-cut shears is 48 ft., and (allowing for the height of the stand pipe at the return tanks) the effective head in the return main is 32 ft. The water in the return main therefore cannot be hurried, and the surge tanks need to be of large capacity to tide over periods of fast working, *e.g.*, when cutting up a scrap plate for return to the melting shop.

RETURN TANKS

In a system where overhead surge tanks are connected to the return main, the height of the return tank somewhat limits the positive head necessary at the pump inlet. The two tanks in the Appleby mill are 12 ft. 6 in. from ground level to the centre-line of the tanks; each tank is 31 ft. 0 in. long × 6 ft. 6 in. dia., giving a head of 9 ft. 8 in. to the centre of the pump inlet.

Although these tanks appear to be large, from high-water level (1½ ft. from the top of the tank) to the top of the outlet pipe the capacity is 8360 gal., and this is only a 10½ min. supply for a discharge rate of 800 gal./min. It is therefore necessary to have a reliable float indicator in the pump house. The volume from high-water level to the bottom of the tank is 10,700 gal., and for this flow rate the velocity in the tank is 0·04 ft./sec.; thus the water takes about 13 min. to traverse the tanks, and this, together with the low velocity, gives time for suspended matter to settle. To help to retain this sediment, a baffle of height level with the middle of the outlet pipe is placed about 5 ft. from that end. Each tank is fitted with a large-diameter sludge valve in the bottom, and the low-level cross pipe at the inlet end and the outlet pipes at the opposite end are valved so that one tank can be shut off for cleaning while the plant is in operation. All these valves are of the full-way type.

Entrained air plays havoc in multi-stage pumps, and therefore all return pipes, dosing, and make-up water pipes were modified to have submerged outlets. It was found equally important for the return water pipe from the pump balance valves to have a submerged outlet at the far end of the tank. The volume thus returned is small, but air bubbles may be entrained if it is allowed to splash in near the outlets.

The supply and treatment of water for hydraulic and other systems are dealt with in Appendix II, which also deals with the rate of sludge deposit in the tanks.

PUMP FEED PIPES

Cavitation in the pumps causes water hammer and erosion, and to avoid this the feed water must be able to keep in step with the fluctuating demands of the pumps. Fortunately, these changes are not instantaneous. In a reciprocating pump fitted with a by-pass from delivery to inlet for off-loading, the rate of change is controlled by the speed of the by-pass valve. Similarly, with centrifugal pumps the rate of change depends on the rate of opening of valves in the using system and on the acceleration of water in the main to this system. Nevertheless, return tanks provide only 4·15 lb./sq. in. head in the feed pipe, and there is a strainer at the tank outlet which probably takes ½ lb./sq. in. of this head. The feed pipe from the tanks is 12 in. bore by about 45 ft. overall length, with 6-in. dia. branches to four 5-stage 500-gal./min. centrifugal pumps. With the low head available this system was not very satisfactory, but excellent results were obtained when the main was extended about 15 ft. to a surge tank. During a momentary reduction in demand, the water in the main can continue to flow into the surge tank with slight loss of velocity; it is thus ready for the next upward pulsation. Similarly, a sudden large increase can be supported by a draw-off from the surge tank. This tank was connected by a ring main back to the main tanks, but experience has shown that this is not necessary.

Fig. 6—Pump alignment gauge

When only a small head is available the velocity in the main should not exceed 3 ft./sec., or preferably 2½ ft./sec.; also, the tees should have large branches. In this system a 12 in. × 12 in. × 10 in. branch tee was used, with a 10 in. to 6 in. × 3 ft. long taper piece to the pump inlet bend.

PUMPS

Pumps are the heart of any hydraulic system, and it might not be out of place to mention the steps that have been taken to maintain the Appleby pumps in good condition. These are primarily to have correct neck bush and balance valve spigot clearances and correct longitudinal alignment of the impellers with the diffusion chambers. When the bearing has worn down to half the neck bush clearance, it must obviously be remetalled and bored. Similarly, the fit of the bearing housing spigot in the pump end casing must be exact.

The longitudinal alignment of the impellers is checked by the gauge shown in Fig. 6. In this gauge,

the three feet are placed on the face of the first-stage end casing and a mark on the pump spindle must lie within the scope of the 'setting' and 'maximum wear' (notched) blades.

The impellers in the first two stages were of stainless steel and showed practically no wear; the remaining three were of phosphor-bronze, and their vane tips were badly worn. These were changed to stainless steel, and since then impeller efficiency has remained high and maintenance has been reduced. The diffusion chambers and guide vanes are also in stainless steel.

Since making these modifications the plant has been able to operate at a higher pressure with two pumps instead of three.

ACCUMULATORS

A strategically placed accumulator reduces the acceleration head when a working cycle is started. The higher initial pressure, permitting a smaller working cylinder, reduces the volume of water required, and this further reduces the acceleration head. Alternatively, the smaller acceleration-head loss can be used to speed up the work cycle.

The secondary rôle of the accumulator is to relieve the system of shocks when a valve is closed at the end of a work cycle. For this purpose the air-loaded type is probably better, as only the mass of water in the accumulator has to be accelerated. In the 345-gal. accumulator referred to on p. 274, this mass is 1½ tons, compared with the additional 178 tons of ballast.

In the Appleby system the accumulators are weight-loaded, but as the question of air-loading was also investigated, a comparison will be made.

Weight-Loaded Accumulator

Gland Friction—Published information on gland friction is scanty. A research on pressure, ram diameter, hydraulic fluid, type of packing, and number of rounds in the stuffing box, might well result in valuable improvements in design. Observations made at the mill accumulator showed that the ram would begin to rise when the pressure was 25 lb./sq. in. above the static pressure of 750 lb./sq. in., and would continue slowly at 15 lb./sq. in. above the static pressure, *i.e.*, at 765 lb./sq. in. The pressure gauge was level with the top of the column of water in the accumulator cylinder. When falling, the pressure would continue to fall at or just below 750 lb./sq. in., but from this should be deducted the 15-ft. head of water in the cylinder.

Inertia of the Ballast—The force required to accelerate the ballast is found in the pressure differential between the line and the accumulator. Even at the usual low accelerations this is a considerable quantity.

For any specific flow conditions, the greater the mass of ballast the less will be the pressure difference required to accelerate it, because the larger the ram area the smaller will be the acceleration.

The fundamental equation (p. 271) can be rewritten in terms of pressures:

$$FA = \frac{PAa}{g}$$

and similarly:

$$a = \frac{144Q}{AT},$$

where: F = accelerating force, lb./sq. in.
P = static pressure of accumulator, lb./sq. in.
A = ram area, sq. in.
a = acceleration, ft./sec. sec.
T = acceleration time, sec.
Q = steady flow after time T, cu. ft./sec.

Thus, for unit flow after unit time:

$$F = \frac{144P}{gA}.$$

Thus the pressure intensity to accelerate the ram varies inversely as the ram area, and better characteristics are obtained for any required accumulator volume by using a large-diameter ram and a correspondingly shorter stroke. The large diameter reduces both gland friction and acceleration head.

Work Done by the Accumulator—From observations made with the mill working normally, the downward travel of the mill accumulator is 3–15 in., giving a discharge of 5·8–29 gallons. As shown in Tables I and II, to start the accumulator falling the pressure drops to about 625 lb./sq. in. on receiving a demand, then quickly rises to 715–725 lb./sq. in. with the fall continuing. The average rate of fall is 0·3–0·4 ft./sec. When the demand is satisfied, the pressure rises suddenly to 800–850 lb./sq. in. and the fall is arrested almost at once. The accumulator discharge for various falling speeds, and the time to stop discharge after increasing the pressure, are given in Table III. As soon as the fall is arrested, the accumulator rises and the pressure falls to about 775 lb./sq. in. The average rate of rise is 0·17–0·2 ft./sec.

The average cutting time at a shears is 15–25% of the total time cycle, including running in and setting the plate, so that the accumulator has ample recovery time.

Relief Valve—To overcome internal losses, the accumulator must be loaded to about 25–50 lb./sq. in. less than the normal pump pressure. Moreover, at closure of a control valve elsewhere, the mains pressure rises temporarily about 125 lb./sq. in. above normal. If the accumulator is already at the top of its stroke, this imposes a shock load of about 35 tons on the cross beam. This can rise higher, with serious consequences, unless a mechanically operated relief valve is provided. The relief valve must have a cushion-

Table III

ACCUMULATOR DISCHARGE

(Static load: 750 lb./sq. in.)

Ram Velocity, ft./sec.	Discharge, gal./min.	Gauge Pressure, lb./sq. in.		Stopping Time, sec.
		Stop	Deceleration Pressure	
0·2	276	800	70*	0·06
0·3	414	800	70	0·10
0·4	550	800	70	0·133
0·5	690	800	70	0·166
0·2	276	850	120*	0·04
0·3	414	850	120	0·06
0·4	550	850	120	0·08
0·5	690	850	120	0·10

* Includes 20 lb./sq. in. gland friction

ing action on closure, otherwise violent valve hammering occurs ; therefore, the passages must be rather restricted, and this causes very high velocities and turbulence. The valve used at Appleby is of the ordinary lever and weight-loaded type with a stainless-steel plug and seat containing 18% of Cr and 8% of Ni. Figure 7 shows the violence of the attack even on this material ; the walls of the mild-steel valve body are also eroded away. The valve plug was found to be 18% Cr–1·7% Ni, and the low nickel content may have reduced its resistance. The erosion is more pronounced immediately above the wing of the valve, where cavitation probably occurred. The valve is $1\frac{3}{4}$ in. dia. and the lift when the accumulator is at the top of its stroke is $\frac{1}{8}$ in. ; it will pass about 150 gal./min.

Figure 8 shows a modified valve in which the valve and seat faces were hard-faced with a tungsten-carbide alloy, the hard-facing being carried from $\frac{1}{2}$ in. down the wings to 1 in. up the side of the plug and $\frac{1}{2}$ in. down the bore of the seat. The groove between the valve wings was fluted to give a cushioning effect on closure and to deflect the flow off the face of the seat above. This valve gave good results and is being further improved by fitting a hard-faced bush to the body, as shown, but this has not yet been tested.

Accumulator with Reduced Loading—An accumulator loaded to a lower pressure can be used for an auxiliary circuit if required ; *e.g.*, at the slab mill, where the balance cylinders are operated by a 15 in. × 10 ft. stroke accumulator loaded at 600 lb./sq. in. The accumulator is charged through a 1-in. mechanically operated valve, a balance weight on the tumbler lever holding the valve open or closed as required. The accumulator is connected to the mill through a 3-in. opening to a 3-in. dia. pipe reduced to $2\frac{1}{2}$-in. dia. after 6 ft. Both the 1-in. dia. feed pipe and a 2-in. dia. branch to the $1\frac{1}{4}$-in. dia. relief valve are connected to the $2\frac{1}{2}$-in. dia pipe. The accumulator lifts the relief valve $\frac{1}{16}$ in. The weight-loading of the valve is the same as the static pressure of the accumulator, and the valve can thus accommodate itself to the rate of displacement from the balance cylinder.

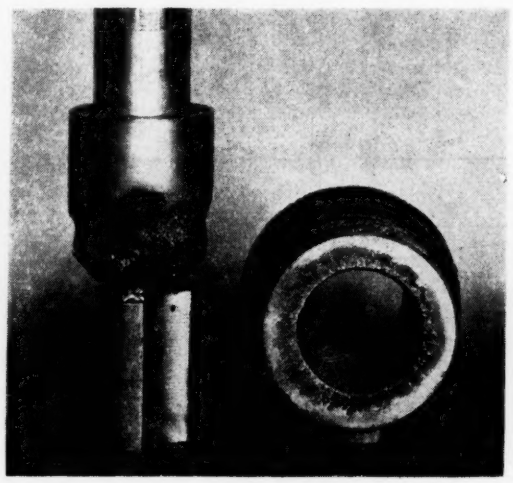


Fig. 7—Valve erosion caused by high-velocity waste discharge

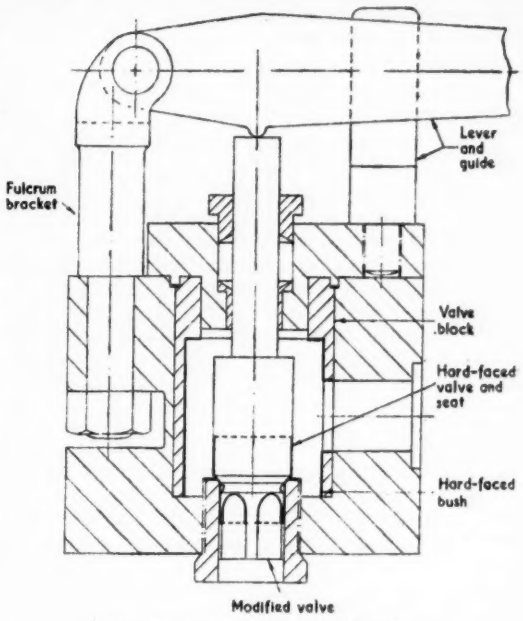


Fig. 8—Accumulator relief valve

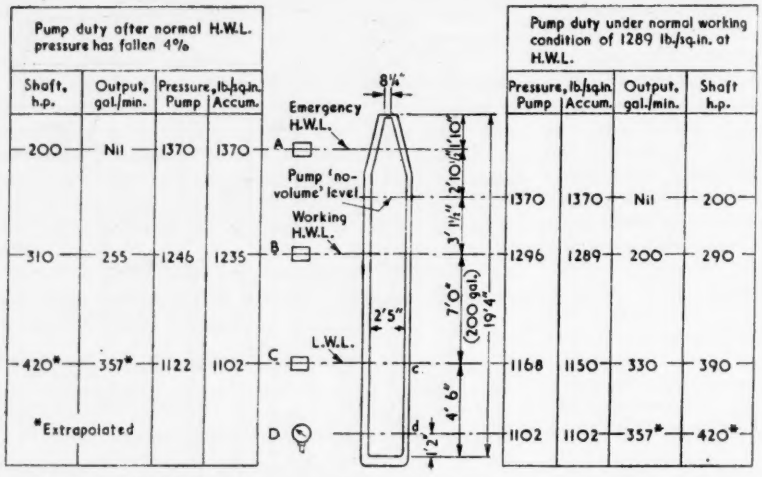
Air-Loaded Accumulator

The air-loaded accumulator has been made possible by the modern techniques for forging and welding steel. The usual arrangement consists of a water vessel and several air vessels connected at the top by a common air-pipe. The characteristic feature of the air-loaded accumulator is that the pressure falls as the vessel discharges, owing to the expansion of the compressed air. The usual practice is to proportion the air and water volumes so that the pressure at low-water level is 10% lower than at high-water level. A smaller range becomes too costly. Figure 9 shows the characteristics of a 200-gal. air-loaded accumulator designed for a proposed descaling plant ; three air vessels were coupled to it, of the same size as the water vessel.

It was proposed to use multi-stage centrifugal pumps on the system. These had falling pressure/volume characteristics, and Fig. 9 shows the pressure and pump output for the high and low level conditions in the accumulator and the rise in level at no delivery head of the pump. The pump should be designed so that its pressure/volume characteristic over the working volume range is similar to that of the accumulator, then each does its fair share of the work.

The air pressure in the accumulator will gradually fall, owing to its solubility in water ; it is also affected by the ambient temperature of the room. To replenish the losses a small 3-stage compressor is required ; this need only be of about 10 h.p. and would probably be used once a week for an hour or so to ' top up ' the pressure.

The M and N water-level controller is shown in



A, *B*, *C* = M and *N* relays
D = Pressure gauge with trip contact in M and *N* circuit. Trips between pressures *c* and *d* dependent on state of pressure at *B*

Fig. 9—Operating conditions for 200-gal. air-loaded accumulator

detail in Fig. 10. A see-saw lever in a small pressure vessel has a steel weight at one end and an aluminium weight at the other. An iron rod connected to it acts as a core for a relay coil, which is fitted outside the body of the vessel, and operates an armature to energize an external relay circuit. When the vessel becomes flooded, the aluminium weight, being of greater volume, is more buoyant ; the steel weight thus overbalances it and moves the core of the relay. This alters the magnetic field and thus causes the armature to make or break the circuit, as required. By reversing the weights the system can be made to work in either direction, and it also has the advantage that there are no packed glands.

In Fig. 9, *B* and *C* are arranged so that the discharge valve to services shuts at low-water level and reopens at high water. After high-water level has been reached, the valve remains open at all levels above low water. The pressure-gauge trip circuit is an extra shut-down precaution against air entering the system. The emergency high-water level relay at *A* is to shut down the pumps if the air pressure should accidentally fall to a level at which flooding of the air cylinders might occur.

When high shock pressures are encountered on valve closure, the accumulator must be operated with a larger margin above the high-water level.

Effect of Air Temperature on Accumulator Pressure —With an accumulator of this size, a fall in the internal air temperature of 20° F. from 80° F. would cause a 3·55% drop in pressure, which is considerable. However, the mass of the steel cylinders is so great compared with the air content that a change in the air temperature would be slow, and could be still further reduced by lagging the cylinders.

After a week-end shut-down in cold weather it

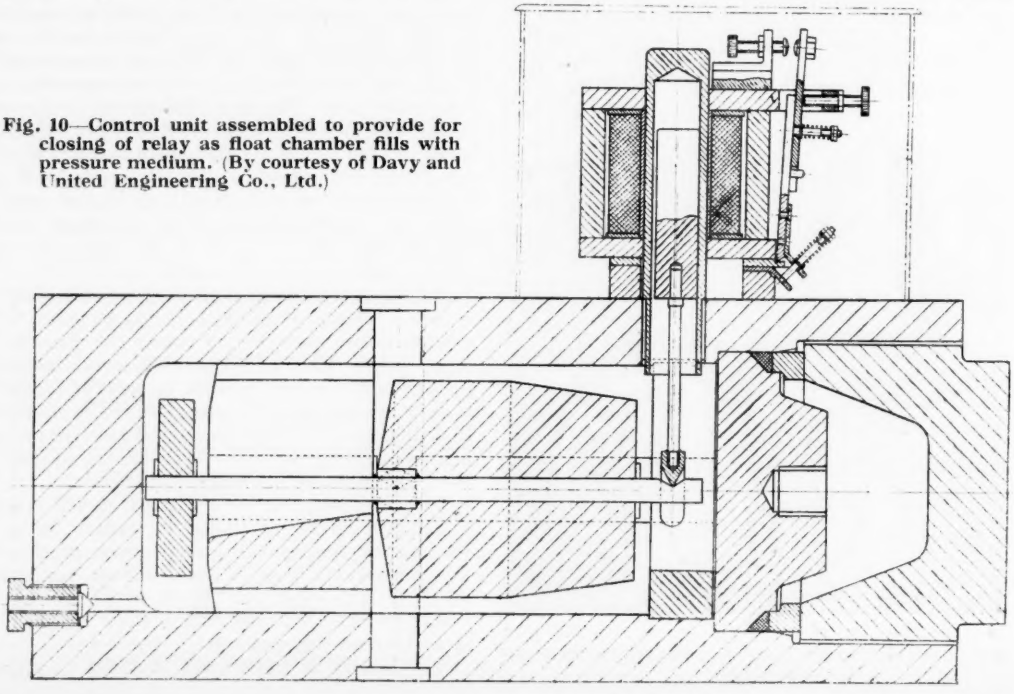
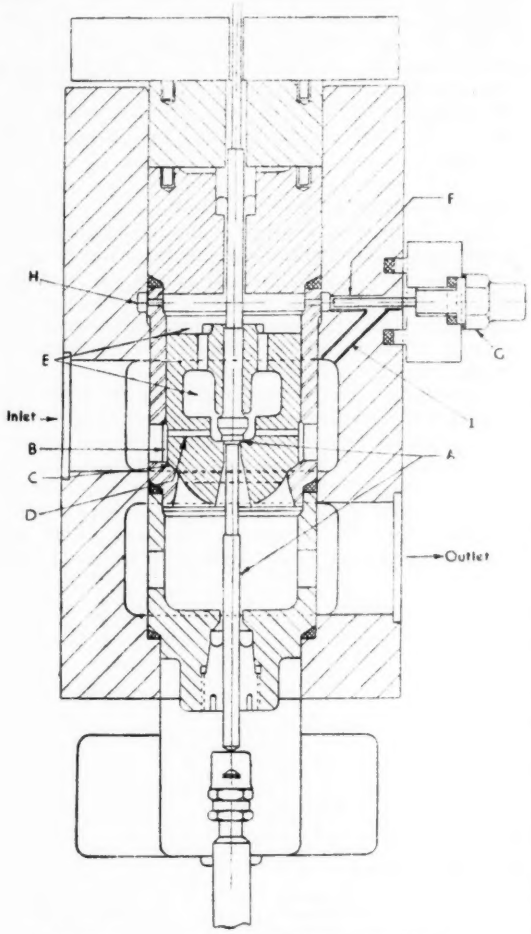


Fig. 10—Control unit assembled to provide for closing of relay as float chamber fills with pressure medium. (By courtesy of Davy and United Engineering Co., Ltd.)

might be necessary to run the compressor to bring the pressure back to normal. During the week, however, warm water is continually entering and leaving the accumulator, and the air is continually undergoing adiabatic changes ; thus the pressure should be little affected by external conditions.

Effect of Solubility of Air in Water—The volume of air absorbed by water to produce saturation varies with temperature but is unaffected by pressure. At 50° F., this volume would be 2·33% of the water volume at atmospheric pressure, or 0·0017 lb. per cu. ft. of water. At 1200 lb./sq. in. pressure, the increase in density of this volume would increase the weight of air absorbed to 0·137 lb. per cu. ft. of water, an increase of 81 to 1. The absorbed air would be released in the return system, and would therefore be a considerable nuisance. In practice, however, the water near the surface of the column in the accumulator remains substantially unchanged, and offers only a small surface area to the adjacent air.



A. Pilot valve
B. Main valve
C. Main valve seat
D. Balance pressure port
E. Pressure chambers
F. Throttling annulus to control valve closing speed
G. Nipple screwed into flange
H. Chambering
I. Passage to inlet annulus

Fig. 11—Pilot-operated control valve

If, with oil at high pressure as the working fluid, there is danger of oil vapour forming an explosive mixture with the compressed air, the air could be replaced by an inert gas such as nitrogen.

HIGH-PRESSURE MAINS AND BRANCHES

All high-pressure mains are of solid drawn steel tubing, with welded flanges having spigot and socket annuli on their faces, and copper gaskets.

Branch pipes up to 4-in. bore are screwed with parallel threads, and the flange of one pipe is screwed back so that the pipe end projects $\frac{3}{8}$ in. to form a spigot. The other flange has the thread cleared $\frac{3}{8}$ in. back from the face so that a spigot and socket joint is formed by the mating ends of the pipes ; $\frac{1}{8}$-in. copper serrated gaskets are used between the ends. This type of joint is cheap, and is easily repaired on site.

Shock Pressures

Alleviators or cushion valves have been found troublesome, and are no longer used. In a large system the end of a cycle in one machine may be the starting point in another, so that flow is either diverted or only partially arrested. Also, the valve closure time on a large machine is 0·3–0·5 sec., and the rising friction head of the valve dissipates much of the energy.

Referring to Tables I–III, the weight-loaded accumulator limits the pressure rise in its vicinity to a reasonable amount, and also provides a channel for some of the water that was being used at a machine. In the air-loaded accumulator, the water level will continually adjust itself to the mains pressure in its vicinity, and thus, as a machine control valve closes, it commences to take in water. Consequently, the back-pressure rises, which in turn reduces the output from the centrifugal pumps, and the whole system either falls to a lower rate of flow or to rest, according to the amount of the remaining demands.

VALVES

Stop Valves

In the screw-down type of valve the spindle should be at 45° with the inlet and outlet flanges, to streamline the flow. These valves are only required in an emergency, so the screws, valves, and seats must be of non-corrodible material.

Control Valves

Control valves usually present to the fluid a rather tortuous path, which may change both direction and area up to six times. Also, the area of the annulus around the valve cage is often too small. These conditions cause a pressure loss and therefore need careful investigation. In a good design the pressure drop through the valve, at an entry velocity of 20 ft./sec., should be 8–10 lb./sq. in.

In valves larger than $\frac{3}{4}$ in. the rate of closure is usually delayed, so as to reduce shock pressures. One type is the pilot-operated valve. This is not only easier to operate against line pressure, but also has an automatic delayed closure independent of the speed of the external operating mechanism. The usual arrangement is shown in Fig. 11. Line pressure is maintained in the chamber *E* by a small hole

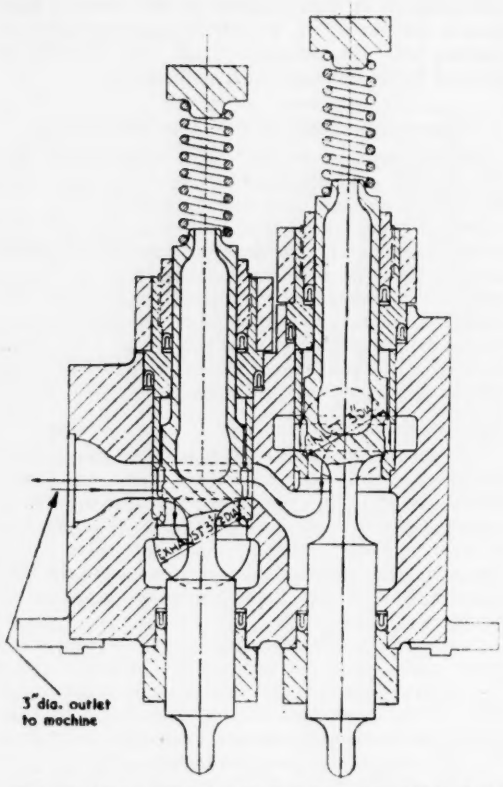


Fig. 12—3-in. direct-acting relay-operated valve

D in the body of the main valve *B*. On lifting the pilot valve *A*, the chamber pressure falls because the pilot valve area is greater than the small feed hole. The pressure load is thus taken off the valve, which can then be lifted easily by the shoulder on the pilot valve. To close, the pilot valve is lowered on to its seat, pressure builds up in chamber *E* through the small hole, and this, aided by an external spring, lowers the main valve. External adjustment of closure speed could be achieved by replacing the small hole in the valve body by the throttling annulus *F* and nipple *G*. The length of the nipple spindle can be altered either to increase or to reduce the stricture. For a single-acting ram the valves are arranged in pairs, one being connected to inlet and the other to exhaust. For a double-acting ram a quadruple assembly is required. Figure 12 shows a direct-acting control valve ; speed of opening and closing is controlled by the shape of the cam on the relay servo mechanism, and also by using a valve with a high lift and a size smaller than the connecting pipe.

For applications such as shearing machines, skid gear, turn-over arms, throw-off tables, etc., such valves provide the quick action needed. For tilting melting furnaces, where a carefully controlled motion is required, a screw-operated '*D*' slide valve is used for reversing and the rate of flow is controlled by the setting of the slide valve or by partial opening of a plug valve on the inlet side. The plug valve is fitted as a safeguard against creep.

Valve Material

The stainless 18% Cr–8% Ni steel used at Appleby for valves internal gives very satisfactory results, except for the relief valves (p. 277). Hard-facing the valve and seat with a tungsten carbide alloy at the mating edges is being tried, to see what further life is possible.

An investigation of the corrosion–erosion resisting properties of various metals has been carried out by A.S.M.E.[1] The tests showed that the wear on 5% Cr–0·5% Mo steel was only $\frac{1}{10}$ of the wear on low-carbon cast steel, and 12% and 18% Cr steels showed respectively $\frac{1}{2}$ and $\frac{1}{3}$ of the wear on the 5% Cr steel. For economic reasons, therefore, it is suggested that the 5% Cr steel should be tried out on hydraulic systems.

Relays and Jacks for Control Valves

One of the simplest relays is the 'thrustor,' in which a 3-phase squirrel-cage motor with remotely operated direct-to-line starter drives the impeller, which builds up pressure behind the piston. This in turn oscillates the valve rocking shaft through the clevis. At the top of its stroke the impeller continues to spin harmlessly in the hydraulic oil.

The 10-ft. mill skid gear, turn-over arms, etc., are operated by a thrustor-actuated battery of 30 double-acting 2-in. pilot-controlled valves. There are two thrustors for each valve coupled through lost-motion links to the valve rocking shaft ; each is of 500 lb. thrust × 5 in. stroke. These thrustors take 2 sec. to complete their stroke against full load, but as they are oversize for the valves, have $2\frac{1}{2}$ in. of travel before

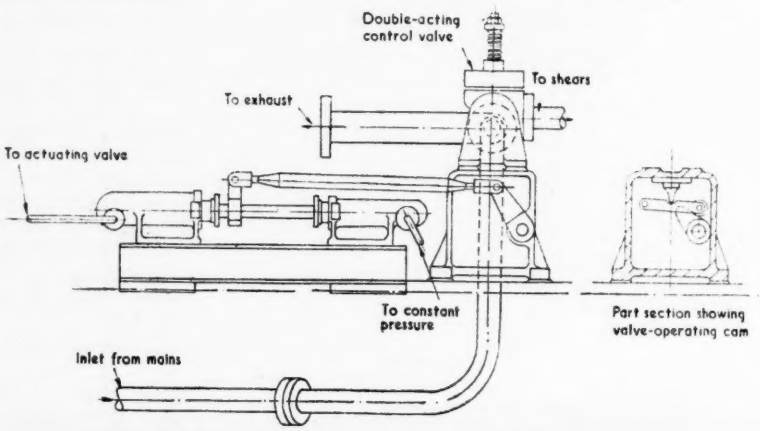


Fig. 13—Hydraulic relay actuated by mains pressure

taking up the load, and do not have to build up full pressure, they are practically instantaneous.

Another relay which is particularly convenient for an isolated machine is one using pressure from the hydraulic main for controlling a valve connected thereto (*see* Fig. 13). There are two rams in opposition, one connected freely to line and the other (of twice the area) coupled through a double-acting poppet valve. The push rod from the ram rod oscillates a camshaft which in turn lifts the valve spindle. In this instance the valve is of direct-acting design (*see* Fig. 12), but the cam of the actuator can be designed so as to retard the valve over the initial and final phases of movement and thus reduce the shock to the machine when starting the cycle and the deceleration head when finishing it. Throttling the rams by means of a small operating valve can also be employed, but this increases the overall time, whereas the cam can be designed to change the shape of the acceleration curve.

This double ram is very convenient when only two conditions are required, such as ' open ' or ' shut.' When a third is needed, such as ' hold,' the two rams should be of equal area and controlled by a quadruple-acting valve. In this, ' mid-position ' closes both inlet and exhaust valves for each ram. Alternatively, a double-acting cylinder can be used in conjunction with the quadruple-acting valve.

The third system is essentially a subsidiary hydraulic service using oil as the working fluid, and comprises a pumping station, air-loaded accumulator stations at focal points, and a mains system between them. It is particularly suitable where a number of diverse operations have to be automatically or manually controlled, and both control methods can be used in the same system. Another advantage of this system is that it operates at a high pressure, so that oil lines, relays, and operating jacks can be of small size.

For relay work there should be a pressure sustaining device, so that when the pump is unloaded against a no-demand period the pressure in the line is retained. A maximum pump pressure of 2000 lb./sq. in. is usual.

The air-loaded accumulators have a static pressure differential of 2000–1500 lb./sq. in. between fully charged and low oil level. A floating piston fitted with synthetic rubber pressure sealing **U**-rings separates the air and oil, and the piston may also be fitted with scraper rings. The **U**-rings seal off the air from the oil when the accumulator is fully discharged, and so prevent air entering the system.

Two sealing rings are necessary, placed back to back, and these will offer a frictional resistance which depends on the pressure. If this loss is assumed to be 3%, for two rings it represents a pressure differential of 180 lb./sq. in. between charging and discharging at a static pressure of 1500 lb./sq. in. Thus, the discharge pressure would be 1410 lb./sq. in. After allowing for line and relay valve losses, 1200 lb./sq. in. may be assumed as the working pressure at the jack. When calculating the jack piston diameter the resistance of both piston and piston rod sealing rings has to be considered. In a double-acting piston, one piston ring will be at exhaust pressure whilst that on the piston rod ring will vary according to whether it is on its pressure or exhaust stroke.

Figure 14 is a diagram of an oil relay system ; two relays are required to operate a double-acting jack, one relay being coupled to the pressure line and the other to the return tank. In the normal position, the shuttle valve needle valve is closed to line pressure by the spring ; thus the servo pistons will be up and relay *B* will connect the pressure line to the right-hand end of the jack, whilst relay *A* will connect the left-hand end to the tank and the jack piston will move to the left. On opening the needle valves, the servo pistons are forced down and pressure conditions in the jack are reversed by the main valves ; the jack thus moves to the right. On closing the needle valves again, the servo pistons automatically rise, because the servo piston in relay *B*, being down, passes pressure to the underside of the servo piston *via* port B_2 and the main valve. This lifts the piston, which is held up by pressure on the main valve. Pressure then passes to the right-hand end of the jack and also by the common pipe *via* port A_1 to the underside of the servo piston of relay *A* ; the piston is thus lifted and connects the left-hand end of the jack to exhaust *via* ports A_3 and A_2.

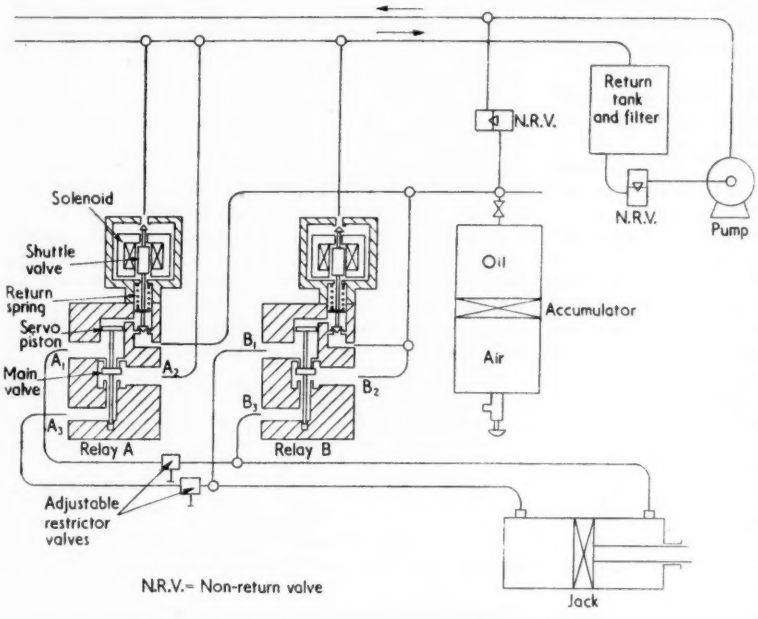


Fig. 14—Oil-operated relay to operate control valve

The speed of the jack in one or both directions can be modified by placing adjustable restrictor valves in the return lines. These build up a back-pressure against the operating pressure.

The shuttle valve is operated by a solenoid and return spring, as shown. The needle valve orifice is 2 mm. dia., so that at 1400 lb./sq. in. the force on the valve is only 6·8 lb.

Any number of jack and relay groups can, of course, be coupled to the system, provided that the pump and accumulator capacities are adequate for the sequence rate. The solenoids can be operated by push-buttons through contactors, or automatically by photo-cell or cam switches controlled by the work or by a time-operated sequence switch.

To the same system can be connected small jacks, operated either by the shuttle valve portion of the relay only or directly by a multi-port semi-rotary slide valve. It would seem also that the servo pistons of the pair of relay valves could be operated directly by one pipe connecting their servo pistons to a manually operated double-ported valve admitting pressure from the line or connected to the tank as required.

For a single-acting jack or one in which the return is by a constant-pressure cylinder connected directly to line, only one relay is required. In Fig. 14, valve chamber A_2 would be connected to the jack and ports A_1 and A_3 to pressure and tank, respectively.

The following points should be noted in designing such a system.

If the system is large, an accumulator should be placed near a group of users so that the velocity in the main from the pumping station is reduced to that of the average demand ; this will allow smaller pipes to be used and will result in steady pressure conditions at the users. The bore of the tubing must be smooth.

For branch lines to relays and large jacks, $\frac{1}{2}$-in. true bore solid drawn steel tubing of $\frac{1}{16}$-in. wall thickness is used, and $\frac{3}{8}$-in. O.D. copper pipe × $\frac{1}{4}$-in. bore, or Bundy tubing, for shuttle valves. The British Ermeto coupling can be used with advantage.

Where flexible hoses have to be used, their length must be generous because they stiffen under pressure.

The self-sealing coupling is a useful fitting. If the apparatus is rapid acting, the pressure drop through the coupling should be found by a bench test before fitting it.

The return tank at the pumps should be the highest point in the return lines, which should if possible have a slight rising gradient, because dissolved air may come out of solution at the low return pressure. A good oil filter is required ; also, to reduce the amount of air dissolved, all return pipes should be submerged below low oil level to avoid turbulence. The air vent should be fitted with an efficient filter. A silica-gel dehumidifier could also be attached to the intake.

HYDRAULIC OIL

Hydraulic oil must be fluid enough to make an efficient medium for the transmission of power, yet at the same time it must possess sufficient viscosity to act as a good seal and to be a good lubricant. It should preferably have a high viscosity index, *i.e.*, the change of viscosity with temperature should be low. It must be chemically stable so that the same oil can be used for long periods without oxidation. It must also be anti-foaming and must readily separate from water.

Oxidation is greatly delayed by the addition of an inhibitor, but some moisture becomes entrained and this, in conjunction with dissolved air, will attack ferrous surfaces, the rust so formed acting as a catalyst of oil oxidation. The oil must therefore have good wetting properties, to prevent water from coming into contact with metal surfaces, and should contain a rust inhibitor. Foaming, caused by the release of air, can be reduced by the addition of a suppressant. These properties which increase stability thereby increase demulsibility, which in turn reduces the rate of undesirable changes.

The required viscosity of the oil depends to a large extent upon the pump used. In a system in which the pump is directly connected to the user by a short pipe, transmission loss resulting from high viscosity is not important. On the other hand, in a system such as that described, where the oil is used for relays and jacks operating auxiliary mill services, the oil has to be pumped long distances, so that equipment suitable for oil of medium viscosity is more economical in both initial and running costs. For this system an oil of viscosity 200 Redwood I sec. at 70° F. was used ; Fig. 15 shows the relationship between flow, velocity, and friction head. At this viscosity the flow is streamline for the $\frac{1}{2}$-in. and $\frac{3}{4}$-in. pipes covered by the scope of the graphs, but for 1-in. and $1\frac{1}{2}$-in. pipes at velocities greater than 13·6 and 9 ft./sec., flow enters into the transition stage between streamline and turbulent flow, and friction losses over these portions will fluctuate between the dotted and chain-dotted lines for semi-turbulent and streamline flow. Poiseuille's formula for streamline flow is given in Appendix I. The dotted curves representing semi-turbulent flow were calculated in the same manner as the friction-head curves for water in Appendix I, the friction factor being taken from the dotted '*S*' portion of the Stanton curves referred to therein.

The quantity of oil used in relay jacks for valves, etc., is small, but the rate of usage is high, so that the sizes of connecting pipes must be considered. As a practical example, a 3 in. dia. × 4 in. stroke jack is used to operate a 4-in. hydraulic valve ; the total time to open the valve is 0·4 sec., of which 0·2 sec. is accelerating time. The pipe connecting the relay to the jack is $\frac{3}{4}$ in. bore × 30 ft. long, and the relay would be within a few feet of the accumulator. Only 0·1 gal. of oil is used, but the final rate of flow is 20·4 gal./min. and applying the above formula the following information is given :

Pressure difference through relay ...	30	lb./sq. in.
Acceleration head	31	,, ,,
Friction head	30	,, ,,
Entry and exit losses (say)... ...	10	,, ,,
	101	,, ,,

Similar losses causing a back-pressure will occur in the exhaust line and exhaust relay from the other end of the jack. These losses must be deducted from the minimum pressure exerted by the accumulator, and when calculating the jack piston diameter an

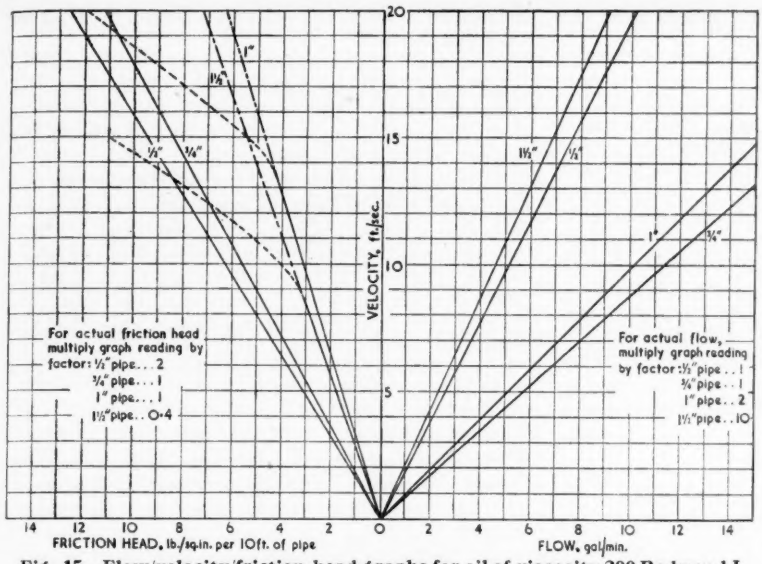

VELOCITY, ft/sec.
FRICTION HEAD, lb./sq.in. per 10ft. of pipe
FLOW, gal/min.
For actual friction head multiply graph reading by factor: ½" pipe... 2
¾" pipe... 1
1" pipe... 1
1½" pipe... 0·4
For actual flow, multiply graph reading by factor: ½" pipe... 1
¾" pipe... 1
1" pipe... 2
1½" pipe... 10

Fig. 15—Flow/velocity/friction-head graphs for oil of viscosity 200 Redwood I sec. at 70° F. per 10 ft. run of pipe

allowance must be made for piston and rod packing ring friction.

The return pipe from the relay cabinet to the pumping station may involve 180 ft. of 1½-in. piping, and the discharge from this jack would cause the following losses :

Acceleration head	47	lb./sq. in.
Friction head	11	,, ,,
	58	,, ,,

It is not possible to use a surge tank, as was done for water from the hydraulic shears (p. 274), since this would expose the oil to further contact with air. However, a low-pressure air-loaded accumulator with a floating piston is feasible. At 10–15 lb./sq. in. the accumulator would receive exhaust oil from all local sources with only its back-pressure loss, and would pass the oil forward at a steady average rate involving a friction head of only about 5 lb./sq. in.

MAINTENANCE

Pump wear is almost negligible because of the oiliness of the hydraulic fluid. Synthetic rubber packings appear to suffer only slight deterioration from the oil. It is important to see that glands do not leak, either from wear or from using the wrong type of oil, since an internal leak in the system engenders heat and this aggravates the leakage by reducing viscosity.

Oil and air filters should be inspected frequently[2] ; the oil strainer should be of 60-mesh wire gauge, and should be three or four times the area of the suction pipe. In addition, a by-pass filter should be fitted, of either the mechanical or chemico-mechanical type, arranged so that the whole contents of the system nominally pass through the filter every 8 hr. The solvent used to remove gummy deposits from pipes and valves may be mixed with the old oil,[2] or the system may be charged with it after draining off the old oil. Since it is circulated by the pump, it must possess adequate lubricating properties.

DESCALING SPRAYS

Considerable information has been published, notably by Holveck[3] and Walquist,[4] on the technique of scale removal from hot slabs and partly rolled plates by high-pressure water jets.

In action, the force of impact of the jet dislodges the scale already loosened by the cooling effect of the water and by rolling. The most effective jet has been found to be a thin flat knife-edge of fan shape, which saves water and excessive cooling, and much experimental work has been done to find an efficient nozzle for producing this type of jet. The required force of impact is 5–10 lb. per inch width of slab, and since this force is a function of volume and velocity, a high pressure is required to produce a greater dispersion and at the same time retain the force of impact. For mild steel this pressure is 1000–1200 lb./sq. in., and for alloy steel it is 1400–1500 lb./sq. in.

The included angle of the jet is about 30°, and the header is usually inclined so that the axis of the jet is at 15° with the normal to the slab. The centre distance between nozzles should allow for ½-in. overlap of the jet edges at contact with the slab, and the nozzle is set in the header so that the flat surface of the jet is twisted 15–45° with the centre-line of the header. This twist prevents interference at the fringes of the jets, and, as the angle increases, there is a greater concentration of impact per inch of transverse width of the slab with the above jet ; however, it is the author's opinion that the twist should be restricted to 15°, as with larger angles the forward wash of the spent water along the slab surface will interfere with the impact of the adjacent jet. For angles of 30° spread and 15° inclination :

$$\text{Transverse coverage} = 0{\cdot}55h\cos\alpha - x \quad \ldots\ldots(1)$$

where: h = vertical height of the nozzle face from the slab
α = angle of twist
x = transverse overlap.

The nozzles used in tests carried out at the Appleby plate mill were of the usual type, in which the perimeter changes smoothly from a circular inlet to a rectangular outlet. Some results are as follows :

Dimensions of Outlet, in. × in.	Area, sq. in.	Pressure, lb./sq. in.	Discharge, gal./min.	Discharge Coefficient
$\frac{3}{8} \times \frac{3}{64}$	0·0175	750	13·6	0·895
$\frac{3}{8} \times \frac{1}{16}$	0·0235	750	20·5	1·01
$\frac{7}{16} \times \frac{1}{16}$	0·0273	725	22·3	0·961

Nozzle inlet diameter = $\frac{15}{64}$ in.
Nozzle length = ½ in.
Included angle of jet for ⅜ in. × $\frac{1}{16}$ in. outlet = 28°.

The discharge coefficients were obtained from the standard formula :

$$Q = \frac{CA}{144}\sqrt{2gh},$$

where: Q = discharge, cu. ft./sec.
C = coefficient of discharge
A = nozzle area, sq. in.
h = head, ft.

This formula can be rewritten :

$$\text{Discharge (gal./min.)} = 31{\cdot}6\,CA\sqrt{P} \quad \text{.....(2)}$$

where: P = pressure in lb./sq. in. at entry to nozzle.

The published discharge data for the Aldrich nozzle with a $\frac{1}{2}$-in. long by $\frac{1}{16}$-in. wide outlet gives a rather higher discharge coefficient.

The force exerted by the jet is given by the formula :

$$F = \frac{WAV^2}{g} \quad \text{.....(3)}$$

where: F = impact force, lb.
W = wt./cu. ft. of water = 62·4 lb.
A = area of jet, sq. ft.
V = velocity, ft./sec.

In this expression, WAV is the weight discharged, expressed in lb./sec. Now using Q as the volume rate of flow, gal./min., C as the discharge coefficient, and transposing P for head, equation (3) becomes :

$$F = 0{\cdot}0631\,QC\sqrt{P} \quad \text{.....(4)}$$

Combining equations (2) and (4) :

$$F = 1{\cdot}99\,AC^2P \quad \text{.....(5)}$$

Allowing for the angle of inclination (θ) of the jet with the slab, equations (4) and (5) become :

$$F = 0{\cdot}0631\,QC\sqrt{P}.\cos\theta \quad \text{.....(6)}$$

$$F = 1{\cdot}99\,AC^2P.\cos\theta \quad \text{.....(7)}$$

where F is now the impact force normal to the slab surface.

From equations (1) and (6) or (7), the vertical height from the slab and the centre distance between nozzles can be calculated. As an example, for the following conditions :

Pressure at nozzle inlet	950 lb./sq. in.
Outlet area at nozzle inlet	$\frac{1}{16}$ in. × $\frac{1}{2}$ in.
Discharge coefficient	0·95
Nozzle inclination	15°
Nozzle twist	15°
Jet overlaps	$\frac{1}{2}$ in.
Impact force normal to surface ...	9 lb.

the discharge per nozzle is 29·2 gal./min., the vertical height of the nozzle outlet from the slab surface is 12·25 in., and the centre distance between nozzles is 6 in.

For complete treatment, nozzle headers are placed above and below the slab, and the upper one must be movable so as to (i) maintain the correct distance from slabs of varying thickness, (ii) be out of the way of turned-up plate ends when not in use, and (iii) give the roller a clear line of vision. Condition (i) can be achieved by attaching the header to a cross-beam connected to the roll neck bearing housings, and conditions (ii) and (iii) by connecting the nozzles individually to the header by radial pipes and oscillating the header to bring the pipes to a horizontal position when not in use.

A canopy must be fitted above the top header to catch water from the bottom header, which does not strike the slab. Also, a fringe or curtain of light chain should be hung along the front and sides to the canopy, to catch flying debris.

To economize in the use of water, the header can be divided into two groups comprising a centre one and two outers. The outer group has a relay-operated isolating valve in the header, and the whole can be operated by pre-selector push buttons which determine the number of groups and also bring the nozzles into the spraying position. This sets the contactor of a relay circuit to the main valve, and the circuit is completed by an automatic switch controlled by the work.

Washing Sprays

In the final passes, loose dust and scale are removed by washing sprays using the same water. These are in a fixed header above the plate only, the nozzles being at 2 ft. 0 in. centres and 4 ft. 6 in. above the work. The water consumption is small, so the valve jack can be manually operated remotely by a two-position oil valve. The water valve should be placed near the header.

POWER APPLICATIONS

Recently, there has been a tendency to use electric power for driving manipulators, shearing machines, and so on. These applications are complicated, since they must always involve a large-ratio gear train and generally have to convert rotary into straight-line motion. Also, an elaborate control gear is required for the continual starting of the motor. 'Stripping down' for maintenance, therefore, is a considerable undertaking.

On the other hand, present multi-stage centrifugal pumps are highly efficient and give immediate response to the varying demands on them. The power unit itself, *i.e.*, the cylinder and ram, is of the simplest form, and only needs the occasional repacking of a stuffing box ; it gives little trouble, particularly if the ram is faced by a chromium deposit producing a hard mat surface.

Balance Cylinders

One of the most valuable uses of hydraulic pressure is the balancing of heavy masses of machinery, since, compared with the mass it balances, hydraulic pressure possesses practically no inertia. Also, its mechanism is simple and occupies little space. It is employed in both hydraulically and electrically driven mechanisms ; *e.g.*, the top roll assembly of a rolling mill, this being held up against the electrically driven screw-down gear by the hydraulic balance cylinders.

It is important that the pressure should be reasonably steady, as if it is too high there is undue wear on the screw-down gear, and if there are large pressure drops the top roll assembly will fall, with serious results (*see* p. 271). Table I shows an example of the amplitude of fluctuations when the sub-station pressure gauge was near the 10–12 ft. mill take-off. Subsequent improvements to the pumps reduced the

pressure drop, but to eliminate acceleration and deceleration head the balance cylinders were connected directly to the 10-in. pump manifold by an existing 4-in. dia. main. This had been initially installed for washing sprays, which were transferred to the general service main.

To find the pressure required to enable the roll assembly to rise in contact with the screw of the screw-down gear, a pressure gauge was fitted at the 7-ft. finishing mill and pressures were taken at which the roll freely rose and fell. With the driving spindle disconnected, the rising force exerted by the balance rams must be 6–7% greater than the supported weight.

The balance cylinder of the driving spindle was also tested with the articulation both horizontal and vertical, and it was found that it may be stiffer in one direction than the other. This is not important in a finishing mill; in a roughing mill, however, where there is a big variation in the angle of drive as the slab is reduced, a stiff articulation causes undue wear both of itself and of the driving gear.

PLANT AT THE APPLEBY-FRODINGHAM STEELWORKS

Melting Shop

Some of the tilting furnaces are hydraulically fitted; they tilt on a circular track of 15-ft. rad., 15° forward to the charging platform for slagging and 35° backwards for tapping. At each end of the furnace there is a double-acting oscillating cylinder of 24 in. bore × 14 ft. 0 in. stroke. High-pressure water is passed to the hollow trunnions through glands and thence by external pipes to the top and bottom ends of the the cylinder, one pipe coming from each trunnion. The piston head is fitted with 2 Lockwood and Carlisle piston rings, $4\frac{1}{2}$ in. wide overall, and connects to a 9 in. dia. × 18 ft. 0 in. long piston rod.

The valve arrangements have already been described (p. 279).

Slab Mill

18 in. × 54 in. 'Up-Cutting' Slab Shears—The central 36-in. dia. ram brings the top blade cross-head and clamping plate down to the slab and grips it against a stool underneath. The ram cylinder is in a large entablature, which is connected to the bottom blade cross-beam by two 21-in. dia. rods. Thus, when the top blade is arrested by the clamping action, the entablature has to rise under the pressure in the cylinder and carry out the cutting stroke by means of the bottom blade.

Power for the cutting stroke is provided by a steam-operated intensifier, which takes water from an overhead tank and delivers it to the 36-in. dia. ram cylinder at $2\frac{3}{4}$ tons/sq. in.

After the cut, the control valve opens the water system to exhaust into the overhead tank, the entablature and connected bottom blade fall against a stop, and the top blade cross-head is lifted by two 14-in. dia. × 26-in. stroke rams, operated from the general service hydraulic main, until it is also against a stop. This retracts the 36-in. dia. ram into the cylinder in the entablature. At the same time, the intensifier ram falls and takes water from the tank ready for the next cut. The intensifier has a 7 ft. 3 in. dia. × 9 ft. 6 in. stroke steam cylinder, and a 14-in. dia. hydraulic ram coupled to it by two piston rods and an underslung cross-head. The steam pressure is 160 lb./sq. in.

The displacement of the intensifier will cut slabs up to 10–12 in. thick; for thicker slabs a holding valve is operated, which retains the pressure in the 36-in. dia. shear cylinder while the intensifier is being recharged.

The main control valve is operated by a steam cylinder from a relay, and the stroke of the intensifier is controlled by a ramp bar.

Alongside the top blade on the entry side of the machine is another clamp bar held by a 9-in. dia. ram concentric with the 36-in. dia. ram and freely connected to the cylinder by a hole up the 36-in. dia. ram. This controls the incoming slab by gripping it against the bottom blade as cutting is in progress, and under pressure it is free to move up with the blade.

Plate Mill

10-ft. Roughing Mill Centring and Turning Gear—The continuous slab reheating furnace has automatic controls of the hydro-kinetic type. The single-acting ram of the centring gear at the mill has a constant-pressure return ram in tandem. The centre dollies for turning a slab 'end-for-end' are raised by a hydraulic cylinder.

Cooling Bank Turnover Arms—In this mechanism there are two 12-in. dia. shafts at $31\frac{3}{4}$-in. centres; each has eight arms secured by two $2\frac{1}{4}$-in. wide keys at 120° in the arm base. The base is split and has a keeper strap secured with four $1\frac{3}{4}$-in. bolts. Each shaft is oscillated by a ram of 18 in. dia. × 4 ft. $4\frac{3}{4}$ in. stroke. The ram cross-head connects through two 12 ft. 0 in. long connecting rods to two 35-in. rad. cranks keyed to the shaft. The 18-in. dia. ram is hollow and is fitted with a 6-in. dia. fixed ram. The fixed ram is also hollow, so that by passing water down it the motion of the main ram is reversed and the arms are lowered. There is also an 'inching' gear which pushes the plates forward if they fall under the turn-over arms.

The plates are skidded sideways by transporter chains, which lie in troughs below rail level. In action, the troughs are lifted by a hydraulic ram through a crankshaft. The crank on the shaft has a roller at the top which lifts the trough in vertical slides. The crank is connected to similar trailer cranks through tie rods, there being a pair of cranks for each trough.

Shearing Machines—The two electrically driven side-trimming machines are fitted with hydraulic clamps and an apron balance cylinder. The latter holds the apron up at the top of the return stroke and automatically declutches the drive to the main crankshaft. Electric manipulators reset the plate after each cut.

In the 2 in. × 12 ft. cross-cut shears the dip table, which carries the incoming plate and allows it to fall clear of the apron as shearing proceeds, falls $1\frac{1}{4}$ in. for each inch of apron stroke. The table is 21 ft. $8\frac{1}{2}$ in. long, and the balance ram at the front end is $7\frac{1}{4}$ in. dia. The rack rolls are chain-driven from the preceding rack.

There are two $19\frac{1}{2}$ in. dia. × $48\frac{1}{2}$ in. stroke cutting

rams connected to 77-in. rad. cranks on the main shaft, and the apron is coupled to 27¾-in. rad. cranks, which give a movement ratio of 2·78 : 1. The apron lifting cylinder is 18½ in. dia. The cutting angle is 1 in 15. Clamps and manipulators are hydraulically operated, and control is by the 3-in. direct-acting valve operated by mains relay rams as in Figs. 12 and 13.

Throw-off Tables—There are three two-way tables beyond the 2 in. × 12 ft. cross-cut shears in the 10-ft. mill, and two single-way tables in the 7-ft. mill. Their action is simple ; the table top has three trunnions on each side which rest in open top bearings on stools. Below, on each side, is a hydraulic ram connected to a crankshaft. This has three cranks with connecting rods to bearing pins in the table frame. The rams are 11 in. dia. × 3 ft. 6 in. stroke connecting to a 2 ft. 6 in. crank. The lifting cranks are 5-ft. rad. and lift the table 22°.

Section Mill Hot Saw

This machine is for cutting rolled sections to length as they come from the Frodingham section mill, and it works in conjunction with a stop and measuring machine beyond the saw which determines the length of the cut piece. The machine is an example of a direct-coupled application using oil as the working fluid. The saw is driven by a 160 h.p. × 734 r.p.m. motor, through an endless belt, the saw speed being 1037 r.p.m. It has a 48-in. stroke and is traversed by two 3½-in. dia. rams at a working pressure of 800 lb./sq. in., the return stroke being by means of a 2¾-in. dia. ram. Oil is supplied by a pump at 68 gal./min. A direct-acting manual valve controls the saw. The cutting speed is 16·35 in./sec. and the return speed is 32·7 in./sec. ; the moving parts weigh 10 tons, so this is a very considerable achievement.

Acknowledgments

The author thanks the Management of the Appleby-Frodingham Steel Company for permission to publish this paper. Thanks are also due to Mr. N. H. Turner, Head of the Engineering Research Department, for helpful criticism ; to various members of the staff, for the preparation of drawings and photographs ; and to the works' engineers, for their co-operation.

The author is indebted to the Davy and United Engineering Co., Ltd., Sheffield, and to Electro-Hydraulics, Ltd., Warrington, for permission to use drawings of equipment normally supplied by them.

APPENDIX I

Derivation of Acceleration Head Formula

Let
F = force, lb.
M = mass, lb.
a = acceleration, ft./sec. sec.
V = final velocity, ft./sec., at end of time T
T = accelerating time, sec.
Q = final rate of flow, gal./min.
A = area of pipe, sq. in.
L = length of pipe, ft.
P = acceleration head, lb./sq. in.

From the fundamental formula $F = Ma/g$:

$$PA = \frac{M}{g} \cdot \frac{V}{T}$$

But
$$M = \frac{62{\cdot}4\,AL}{144}$$

and
$$V = \frac{144Q}{6{\cdot}24 \times A \times 60},$$

Thus, by transposing, the acceleration head is given by :

$$P = \frac{0{\cdot}0052\,QL}{AT}.$$

Calculation of Friction-Head Graphs (Fig. 2)

The graphs for turbulent flow were calculated from the Reynolds formula :

$$P\ (\text{lb./sq. in.}) = 0{\cdot}0556 \left(\frac{R}{\rho v^2}\right) \frac{L}{D} \cdot \frac{\rho v^2}{2g}$$

for water at 50° F. and with kinematic viscosity = 0·000014 f.p.s. units, where D = dia. of bore, ft. ; ρ = density of water, lb./cu. ft. ; v = mean velocity of flow, ft./sec. ; R = frictional force per unit area of wetted surface ; and $R/\rho V^2$ = friction factor, which can be obtained from Stanton curves.[5] These curves connect the friction factor with Reynolds Criterion $\frac{\rho vD}{\eta}\left(=\frac{vD}{\nu}\right)$, where η = absolute viscosity and ν = kinematic viscosity, in f.p.s. units.

When calculating the friction head of the plate mill main, the resistance of valves, bends, tees, etc., was included by the equivalent length of pipe method.[6] Thus, to the actual length of 774 ft. from the pump to the accumulator was added 300 ft. for these fittings.

Poiseuille's Formula for Streamline Flow

Disregarding the negligible kinetic head, the formula for streamline flow is :

$$p = \frac{0{\cdot}067\eta LV}{D^2},$$

where :
p = pressure head, lb./sq. in.
V = velocity, ft./sec.
D = tube bore, in.
η = absolute viscosity, poises.

APPENDIX II

Water Supply and Treatment

By G. W. Cook

The water supply for a works of the size of the Appleby-Frodingham Steel Works is, of course, a large and vital undertaking. About 3·1 million gallons are pumped daily from either the River Ancholme or the North Lincoln boreholes. This water represents make-up, and is about 2¾% of the total water in circulation. The amount of water in circulation is approximately 80,000 gal./min., and this will increase to approximately 130,000 gal./min. when the present extensions are completed.

The make-up for the hydraulic system is 120 gal./min., and this quantity is mainly due to the descaling and washing requirements at the plate mills. Unfortunately, only about 10% of soft water can be supplied in the hydraulic make-up. The remainder is ' hard,'

so that there is a tendency for the excess softening chemicals in the soft water to react on the hard. To reduce gland packing friction and rusting of rams, a tippler dosing gear for adding soluble oil is fitted to the return tanks at the pump house, and the concentration is maintained at 5 parts per 100,000. This represents an addition of 8·7 gal. of oil per day, and this usage has been justified by results.

A point of interest about the make-up water for the hydraulic system is the amount of sludge accumulating therefrom. The water is good, containing an average of 1 grain per gallon of suspended matter, but this amounts to just over 1 cwt. of solids per week of 17 shifts. Some solid will doubtless be carried away by the spray water, but there will also be some temporary hardness thrown down. It was for this reason that the special arrangements (p. 275) for cleaning the return tanks had to be made.

The organic matter in the River Ancholme water has caused trouble in the hydraulic mains, as the warm dark environment of the pipes favours the growth of a thread-like fungus. Attempts have been made to remove this by treating the water with 'chloros' through a dosing gear fitted to the return tanks. It seems, however, that the only successful method would be to render the water inert before using it as make-up : certainly, this trouble is absent when deep well water is used.

The Ancholme river is a large land drain, and the quality of the water is subject to seasonal variations. The total hardness varies between 24 and 33 grains/gal., about half temporary and half permanent, and is almost entirely due to calcium salts. The chloride content fluctuates between 2 and 250 grains/gal., and is occasionally greater. As would be expected from a source of this nature, the organic content is fairly high. The pumps, in the pump house situated on the river bank, deliver the water direct into the works distribution network, a distance of approximately 3½ miles. At the works, 12 million gallons of the river water are held in a reservoir as standby in case of failure of the normal supply. Two remote-controlled pumps, capable of satisfying the demand of the whole works, deliver from this reservoir into the distribution system.

The North Lincoln Borehole water is of good domestic quality, having a temporary hardness of approximately 16 grains/gal. and a permanent hardness of 12 grains/gal., again due almost entirely to calcium salts. The pump house at the head of the boreholes delivers into a 5-million-gallon hilltop reservoir, situated between the source and the works, whence it is supplied by gravity to the distribution mains. North Lincoln water is used for domestic supplies throughout the works, and, after treatment, as feedwater for boilers and locomotives and as make-up to the hydraulic power installations. Temporary failures of the main are covered by strategic connections between the Scunthorpe Corporation and North Lincoln pipes, for the domestic supply, and by an adequate storage of the treated water for the remaining consumers.

The Ancholme water, after removal of temporary hardness with lime, is used for make-up to the cooling systems of blast-furnaces, melting furnaces, gas engines, steam engine condensers, etc. These are on a closed cycle ; each system includes a cooling tower or towers, as a result of which the loss is reduced to 1¾%. The untreated water is used for such purposes as coke quenching, sinter cooling, general washdown, etc.

About 24 tons of chemicals are used each week in the treatment of water from both the River Ancholme and the North Lincoln Boreholes.

Every effort is made towards conservation, and the present arrangements permit maximum re-use of the water. When the quality becomes unsuitable for retention in a particular circuit, the water is bled-off, if possible, into a system where water of a lower quality is acceptable. Only when it is no longer economical to recover the water, owing to its location or poor quality, is it finally discarded.

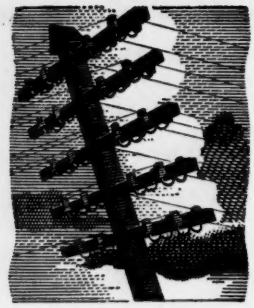
To ensure continuity of the water supply, there is an electrical interconnection between the Ancholme and North Lincoln pump houses, one of which is normally supplied with power from the grid and the other from the works power generating house. This arrangement thus provides both with an alternative electrical supply.

A ring system of distribution mains has been gradually developed. Trunk mains and stop valves are arranged so that, if a failure of a pipe occurs, the section can be isolated in a matter of minutes and supplies can be maintained through an alternative route. To allow this operation to proceed as quickly and smoothly as possible, each valve on the trunk mains and on those leading directly from them has a number corresponding to that shown on the diagrammatic layout of the distribution system. This number and the instructions for operating the valve are cast on the valve pit cover, which is made to an Appleby-Frodingham standard pattern ; it is easily recognizable by those concerned, and is capable of being removed by one person.

For some consumers North Lincoln water is held as standby for Ancholme, and *vice versa*, but to avoid possible contamination of the domestic supplies, the two are not directly connected at any point throughout the system. Arrangements are made for either automatic or speedy manual changeover.

References

1. H. A. WAGNER, J. M. DECKER, and J. C. MARSH : *Trans. Amer. Soc. Mech. Eng.*, 1947, vol. 68, pp. 389–403.
2. R. C. SCHMITT : *Lubricating Engineer*, 1951, Aug., pp. 168–173.
3. J. E. HOLVECK : *Iron Steel Eng.*, 1931, vol. 8, pp. 362–367 ; 1936, vol. 13, pp. 1–10.
4. G. WALLQUIST : *Jernkontorets Ann.*, 1943, vol. 127, pp. 61–89.
5. H. M. SPIERS : "Technical Data on Fuel," 5th Edition, p. 70. London, 1950 : World Power Conference.
6. *Ibid.*, p. 87.



N E W S

ANNOUNCEMENTS AND NEWS OF SCIENCE AND INDUSTRY

IRON AND STEEL INSTITUTE

Annual General Meeting, 1952

The Annual General Meeting of the Institute will be held in London on **Wednesday, 30th April and Thursday, 1st May, 1952.** The opening session will be held at the Royal Institution, Albemarle Street, W.1. After formal business and the submission of the Report of Council and Accounts for 1951, the new President, Capt. H. Leighton Davies, will be inducted. His presidential address will be followed by the Sixth Hatfield Memorial Lecture by Professor E. N. da C. Andrade, F.R.S., on "The Flow of Metals."

Technical discussions will follow, in the afternoon, at the offices of the Institute, 4 Grosvenor Gardens; the papers will deal with the Deformation of Metals.

On the second day discussions will be held at the offices of the Institute on Open-Hearth Furnaces and on Hydrogen in Steel.

Buffet luncheons will be provided on both days at the Institute's offices. The Dinner for members will be held at Grosvenor House, Park Lane, on Wednesday evening.

The full programme for the meeting will be published in the April issue of the *Journal*.

NEWS OF MEMBERS

▶ Mr. W. J. CHADDER, Head of the Coke Oven Sales Department of the Woodall-Duckham Vertical Retort and Oven Construction Company (1920) Ltd., has been appointed a Director of the Company.

▶ Mr. V. GRIFFITHS has taken up an appointment as Research Assistant in the Metallurgy Department of the Massachusetts Institute of Technology, U.S.A.

▶ Mr. W. H. LEWIS, who, as reported in the January issue of the *Journal*, is now Managing Director of Midland Rollmakers Ltd., Crewe, is also Joint Managing Director of the British Rollmakers Corporation, Ltd., Crewe, and Director of the three subsidiaries of Midland Rollmakers Ltd.

▶ Mr. J. A. LOTT has left the British Iron and Steel Research Association to take up an appointment with the Loewy Engineering Co., Ltd., Bournemouth.

▶ Mr. H. M. RYAN has left the Consett Iron Works, Co. Durham, to join the staff of the Blast-Furnace Department of Messrs. Ashmore, Benson, Pease and Co., Ltd., Stockton-on-Tees.

▶ Mr. M. N. SAXENA has completed his training course at the Kulti Iron Works of Messrs. Martin Burn, Ltd., and has been appointed Lecturer in Metallurgy at Roorkee University, India.

▶ Mr. THOMAS H. WILDE has been appointed Vice-President and Director of the Electric Furnace Products Company. He will reside in Bermuda.

▶ Mr. H. CARRINGTON WOOD, Home Sales Manager of The Wellman Smith Owen Engineering Corporation, Ltd., London, has retired after an association of more than 42 years with the organization.

▶ Mr. J. E. WORTHINGTON has left the British Iron and Steel Research Association, Sheffield, and is now on the staff of Thomas de la Rue and Co., Ltd., Warwick.

Obituary

Mr. GERALD C. CLAYTON, Manager of the London Office of the Incandescent Heat Group of Companies, and Director of Controlled Heat and Air, Ltd., on 27th December, 1951.

Monsieur PAUL GIROD, of Ugine, Montmin, France, in December, 1951.

Mr. A. RAMSAY MOON, of Victoria, Australia, on 13th December, 1951.

Mr. H. A. SHAW, Director of Messrs. W. Shaw and Co., Ltd., on 3rd January, 1952.

Mr. HENRY G. STOBART, J.P., for many years Director of Horden Collieries and Wearmouth Coal Co., and formerly President of the Steel Castings Association, during 1951.

IRON AND STEEL ENGINEERS GROUP

Meetings

The **Seventeenth Meeting** of the Group will be held at the Institution of Electrical Engineers, on 4th March at 10.30 A.M.

The **Eighteenth Meeting** of the Group will be held at 4 Grosvenor Gardens, S.W.1, on Thursday, 17th April, at 10.30 A.M.

Particulars of the Meetings were given in the February issue of the *Journal*.

AFFILIATED LOCAL SOCIETIES

Ebbw Vale Metallurgical Society

A Joint Meeting with The Iron and Steel Institute will be held on March 12th, at which Professor W. R. D. Jones, D.Sc., Professor of Metallurgy at the University College of South Wales, Cardiff, will deliver a paper on "*Refractories in Service.*"

Staffordshire Iron and Steel Institute

The Secretary of the new Associate Section for the period January, 1952 to April, 1953 is Mr. R. H. Johnson, 2 Laurel Grove, Penn, Wolverhampton.

BRITISH IRON AND STEEL RESEARCH ASSOCIATION

Staff

Mr. **R. F. Jennings,** who has been Technical Secretary of the Plant Engineering Division since its inception in 1946, has been appointed Head of the North East Coast Laboratories of the Association. He replaces Mr. **E. W. Voice,** who has been appointed Assistant Director (Underground Research) of the National Coal Board, at its Central Research Establishment at Stoke Orchard,

near Cheltenham. Mr. Voice joined the British Iron and Steel Research Association in 1946 as a member of the Field Trials Section of the Iron Making Division. Three years later he was appointed Head of the Section, working on problems of blast-furnace exploration, instrumentation and assessment, sinter manufacture, etc. During 1950 and 1951 he was responsible for the direction of the work of the Section, which had moved to its new headquarters at the North-East Coast Laboratory, Normanby.

The new Technical Secretary of the Plant Engineering Division is Mr. **R. W. Berry**, who has recently joined the Division. He was Technical Secretary of the Steel Castings Division from 1946 to 1950, when its work was transferred to the British Steel Founders' Association.

Mr. **C. N. Kington,** M.B.E., who is Chief Engineer and Local Administration Officer at the Sheffield Laboratories of the Association, has been seconded to the newly formed Cutlery Research Council as its Senior Research Officer. The close link established between the Cutlery Research Council and the British Iron and Steel Research Association will enable Mr. Kington to initiate and develop a research programme under the direction of the Executive Committee of the Cutlery Research Council, but at the same time retaining his present appointment with the Association.

INSTITUTE OF METALS

Dr. C. J. Smithells has been elected President, and Mr. G. L. Bailey and Dr. S. F. Dorey have been elected Vice-Presidents for the year 1952–53.

May Lecture, 1952

The 1952 May Lecture to the Institute of Metals will be delivered by Dr. J. J. P. Staudinger, on "The Place of Plastics in the Order of Matter." The meeting will be held in the Lecture Theatre of the Royal Institution, Albemarle Street, London, W.1, on Monday, 24th March, at 6.0 P.M. Visitors will be welcome; tickets are not required.

NEWS OF SCIENCE AND INDUSTRY

Fifth Empire Mining and Metallurgical Congress

The Fifth Empire Mining and Metallurgical Congress is being convened by the Australasian Institution of Mining and Metallurgy, and will be held in Australia and New Zealand during April and May, 1953.

The chief object of the Congress is to afford an opportunity for mining engineers, metallurgists, scientists, engineers, and others concerned with the mining and metallurgical industries to meet and discuss technical progress and problems, including the development of the mineral resources of the British Commonwealth.

The programme will be divided into five parts: (1) Pre-sessional visit in Tasmania, (2) the inaugural meeting and other official functions, together with technical sessions, in Melbourne, (3) visit to Canberra, (4) further technical sessions and visits in Sydney, (5) one of two excursions to mining and metallurgical centres.

Technical Sessions—The main section of the technical proceedings will take the form of a series of volumes dealing in an analytical and comparative manner with Australian mineral deposits and practices. Papers will be invited from overseas sources on selected specific topics.

Membership—Anyone interested in the subjects to be discussed, irrespective of nationality, may apply for membership of the Congress. Special arrangements will be made for ladies accompanying members of the Congress. Membership fee for the Congress will be £3 3*s.* 0*d.* A member's wife or other near relative will be required to pay an additional membership fee of £1 1*s.* 0*d.*

Copies of the first circular, giving details of the Congress, may be obtained from the Secretary, The Iron and Steel Institute, 4 Grosvenor Gardens, London, S.W.1.

Institute of Physics

The Spring Conference of the Institute of Physics X-ray Analysis Group will be held from 3rd to 5th April in the Chemistry Department, King's Buildings, Edinburgh, in conjunction with the Scottish Branch of the Institute. Further details may be obtained from the Hon. Conference Secretary, Mr. E. G. Steward, Research Laboratories, The General Electric Co., Ltd., Wembley, Middlesex.

Refractory Materials

The Department of Coal Gas and Fuel Industries with Metallurgy, University of Leeds, in association with the Department of Adult Education and Extra-Mural Studies, has arranged two lectures on refractory materials, as part of a programme of special courses. The two lectures will be held on 17th March, 1952, at 2.0 P.M. and 3.30 P.M., and will cover the refractory requirements of the carbonizing industries; the principal properties of silica, silaceous, and fireclay bricks; the durability of refractories in carbonizing plant; and a review of progress made with gas-plant refractories during the last 20 years. The fee for the lectures is £1.

Canadian International Trade Fair

The Fifth Canadian International Trade Fair, sponsored by the Canadian Government and dedicated to Canada's International Trade Fair, will be held at Exhibition Park, Toronto, from 2nd to 13th June, 1952. Particulars may be obtained from the Canadian Government Exhibition Commission, Canada House, Trafalgar Square, London, S.W.1.

Aslib Consultant Service

The Department of Scientific and Industrial Research has made a special grant to Aslib to establish a Consultant Service in the special library and information field. This Service, drawing upon the existing resources of Aslib, and backed by new research into information techniques, is now available to advise those who are considering the establishment of special libraries and information services in industrial and research organizations, and to assist the development of existing services. Details of the Service can be obtained from the Director, Aslib, 4 Palace Gate, London, W.8.

Controlled-Density Steel

The Ontario Research Foundation has developed a process and methods for producing finished and semi-finished articles of steel having any desired density and analysis within a very wide range. The process itself is extremely simple. Fine iron ore is made directly into the desired steel shape without melting or any other intermediate step. The dry, fine, prepared ore, generally finer than 60 mesh, is poured directly into suitable moulds, is then reduced at a temperature of about 1150° C. and is finally held for a longer treatment time of several hours at this same temperature. The resulting article has the composition and structure of normal steel except that it contains a predetermined amount of spherical voids, which are not inter-connected. This point is most important, since the cellular structure of the steel allows it to be heated and hot-worked without any great precautions being necessary to prevent excessive oxidation and scaling.

The future applications of this new engineering material have been divided into three classes, according

to density. The density of steel is about 7·8 g./c.c. Controlled-density steel can be produced with any desired density from about 1·0 to 7·2, directly from the furnace.

Hot-worked Material—Controlled-density steel may be produced with carbon contents ranging from less than 0·01% to 1·5%. Alloying addition can also be made. For hot-working, the density of the steel coming from the furnace must be more than 3·0 and is preferably over 4·0.

Substitute for Iron Powder—Controlled-density steel in the range 4·0–6·5 can be produced with about the same mechanical properties and composition as pressed iron-powder products. In this field the process is in direct competition with powder metallurgy.

Light Steel—In the range 4·0–1·0, the main advantages of the material are the very low cost compared with other light-weight metals, and the fact that it can be produced directly from the furnace to reasonably close tolerances.

The work has been conducted under the aegis of one of the Advisory Committees of the Ontario Research Council and has been supported by funds from the Provincial Government of Ontario.

DIARY

3rd Mar.—CLEVELAND INSTITUTION OF ENGINEERS—"*Engineering Aspects of Open-Hearth Steelplants*," by J. A. Kilby—Cleveland Scientific and Technical Institution, Corporation Road, Middlesbrough, 6.30 P.M.

4th Mar.—IRON AND STEEL ENGINEERS GROUP—Discussion on Accident Prevention—Institution of Electrical Engineers, Savoy Place, London, W.C.2, 10.30 A.M.

4th Mar.—SHEFFIELD METALLURGICAL ASSOCIATION—"*Some Recent Developments in the Use of Metals at High Temperatures*," by J. M. Robertson—Grand Hotel, Sheffield, 7 P.M.

5th Mar.—MANCHESTER METALLURGICAL SOCIETY—"*The Surface Structure and Friction of Metals*," by A. J. W. Moore—Engineers' Club, Albert Square, Manchester, 6.30 P.M.

6th Mar.—LEEDS METALLURGICAL SOCIETY—"*Recent Research on Aluminium and its Alloys*," by D. C. G. Lees—Chemistry Department, The University, Leeds, 2, 7 P.M.

6th Mar.—INSTITUTE OF METALS (Birmingham Local Section)—"*The Steel Company of Wales*," by W. F. Cartwright—James Watt Memorial Institute, Great Charles Street, Birmingham, 7 P.M.

6th Mar.—STAFFORDSHIRE IRON AND STEEL INSTITUTE—"*Government Steelworks of Pretoria*," by N. Dean—"Star and Garter" Hotel, Wolverhampton, 7.30 P.M.

7th Mar.—INSTITUTION OF PRODUCTION ENGINEERS (West Wales Sub-section)—"*Corby Iron and Steel Works of Stewart and Lloyds Ltd.*," by E. A. Taylor—Central Library, Alexandra Road, Swansea, 7.30 P.M.

7th Mar.—WEST OF SCOTLAND IRON AND STEEL INSTITUTE—(Joint Meeting with Iron and Steel Engineers Group)—"*The Abbey Works of The Steel Company of Wales Ltd.*," by R. W. Evans—39, Elmbank Crescent, Glasgow, C.2, 6.45 P.M.

12th Mar.—EBBW VALE METALLURGICAL SOCIETY—(Joint Meeting with The Iron and Steel Institute)—"*Refractories in Service*," by Professor W. R. D. Jones—Lecture Room, Messrs. Richard Thomas and Baldwins, Ltd., Ebbw Vale, 7.15 P.M.

12th Mar.—NORTH WALES METALLURGICAL SOCIETY—"*The Relation Between the Research Worker and the Practical Steelmaker*," by A. H. Leckie—County Primary School, Plymouth Street, Shotton, Nr. Chester, 7.15 P.M.

14th Mar.—SOCIETY OF CHEMICAL INDUSTRY—(Joint Meeting with Association Belge pour L'Etude, L'Essai et L'Emploi des Matériaux)—Symposium on Protective Coatings under Conditions of Atmospheric Corrosion—Iron and Steel Institute, 4 Grosvenor Gardens, London, S.W.1, 10 A.M.

15th Mar.—SWANSEA AND DISTRICT METALLURGICAL SOCIETY—"*Some Recent Developments in the Iron and Steel Industry of America*," by R. H. Parker—Central Library, Swansea, 6.30 P.M.

17th Mar.—INSTITUTE OF METALS (Sheffield Local Section)—Annual General Meeting followed by Joint Meeting with Sheffield Society of Engineers and Metallurgists—"*Bearings and Bearing Alloys*," by R. T. Rolfe—The University, St. George's Square, Sheffield, 7.30 P.M.

18th Mar.—LINCOLNSHIRE IRON AND STEEL INSTITUTE—Annual General Meeting, 7.0 P.M.—G. Elson, 7.30 P.M.—Wortley Hotel, Scunthorpe.

18th Mar.—SHEFFIELD METALLURGICAL ASSOCIATION—"*Development of a British Standard Method*," by J. O. Lay and T. Dennison—Grand Hotel, Sheffield, 7 P.M.

21st Mar.—LIVERPOOL METALLURGICAL SOCIETY—"*Recent Researches on the Mechanism of Solidification in Castings*," by R. W. Ruddle—The Lecture Theatre, Electricity Service Centre, Whitechapel, Liverpool, 7 P.M.

21st Mar.—CLEVELAND INSTITUTION OF ENGINEERS—"*The Measurement of Reheating Furnace Performance*," by F. A. Gray—Cleveland Scientific and Technical Institution, Corporation Road, Middlesbrough, 6.30 P.M.

24th Mar.—INSTITUTE OF METALS—Annual May Lecture—"*The Place of Plastics in the Order of Matter*," by Dr. J. J. P. Staudinger—Lecture Theatre, Royal Institution, Albemarle Street, London, W.1, 6 P.M.

25th Mar.—SHEFFIELD METALLURGICAL ASSOCIATION—"*Metallurgy in University of Sheffield*," by A. G. Quarrell—Grand Hotel, Sheffield, 7 P.M.

25th–27th Mar.—INSTITUTE OF METALS—Annual General Meeting—Park Lane Hotel, London, W.1, 10.30 A.M.

TRANSLATION SERVICE

(The previous announcement was made in the February, 1952, issue of the *Journal*, p. 160).

TRANSLATIONS AVAILABLE

No. 433 (German). E. SIEBEL and S. SCHWAIGERER: "The Strength of Tubes under Internal Pressure at Very High Temperatures." (*Brennstoff, Wärme, Kraft*, 1951, vol. 3, May, pp. 141–143).

No. 434 (German). A. STAERKER: "New Knowledge in the Field of the Surface Protection of Refractory Bricks." (*Tonindustrie-Zeitung*, 1951, vol. 75, No. 3/4, pp. 33–36).

No. 435 (Polish). K. RADZWICKI and J. KOZIELSKI: "Diffusion Deoxidation with Coke, in the Basic Open-Hearth Furnace." (*Prace Głównego Instytuto Metalurgii* 1951, No. 4, pp. 267–277).

TRANSLATIONS IN COURSE OF PREPARATION

(German). H. WENDEBORN: "Recent Developments in Strand Sinter Plants." (Stahl und Eisen, 1951, vol. 71, Nov. 8, pp. 1212–1218).

(German). E. AMMANN and J. HINNÜBER: "The Development of Cemented Carbide Alloys in Germany." (*Stahl und Eisen*, 1951, vol. 71, Oct. 11, pp. 1081–1090).

ABSTRACTS OF CURRENT LITERATURE *and* BOOK NOTICES

IRON AND STEEL MANUFACTURE AND RELATED SUBJECTS

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ORES—MINING AND TREATMENT

Drill Bits with Hard-Metal Inserts and Their Use in Pneumatic Percussion Drilling. (*Maquinas e Metais*, 1951, Sept. 21–26). [In Spanish]. The advantages and economy of using hard-metal inserts are pointed out and some examples of the saving are given. These include the possibility of drilling harder rocks more quickly; elimination of conical boring; eliminating drill changes; use of lighter equipment; and power economy.—R. S.

The Moving-Rung Ore Transporter. J. Zeman. (*Vereinigte Osterreichischer Eisen- und Stahlwerke Jahrbuch* 1950–51, 104–107). An improved design of coal or ore transporter in which steel scraper-bars attached at intervals at both ends to chain drives, move in a flat-bottomed, static trough is described. The transporter is considerably lighter than similar gear of older designs and easier to service. A performance of 120 tons of coal per hour is obtainable.—P. F.

The Sink-Float Ore Dressing Plant at Stripa. P. H. Fahlström. (*Teknisk Tidskrift*, 1951, **81**, Sept. 8, 677–686). [In Swedish]. A heavy-media separation plant of 50 tons/hr. capacity for magnetite with ferrosilicon as medium is described. The purification of the separation medium is discussed, and operational and economic data are given. Finally, a brief description of the Stahlbau Rheinhausen method is given, where the separation takes place in a trough travelling at a speed equal to that of the flow velocity.—B. S. E.

FUEL—PREPARATION, PROPERTIES, AND USES

Oscillatory Combustion in Tunnel Type Gas Burners. D. G. Stewart. (*Indust. Gas*, 1951, **14**, July, 304–310). A theory is suggested to explain the mode of combustion in tunnel-type gas burners. Their high rate of heat release and their high blow-off resistance can be explained by the theory.—D. H.

Systems of Combustion and Burners for Metal-Making Furnaces. H. Schramm. (*Ing. e Indust.*, 1951, **19**, Apr., 85–89; June, 83–88). [In Spanish]. Developments leading to the use of compressed air and control of the gas–air mixture in burners are discussed. The author confines himself to describing the types of combustion system and burner manufactured by The Surface Combustion Corp. Atmospheric, and high- and low-pressure systems are dealt with.—R. S.

A New Mini-Gas Calorimeter. R. P. Donnelly and R. Broadbent. (*Fuel*, 1951, **30**, Aug., 178–181). The construction of a new (Mini) calorimeter is described. It comprises a soap film flowmeter and a simple type of heat exchanger in which the gas is burnt in oxygen and exchanges heat with water, the rise in temperature of which is measured. The calorimeter uses only 200 ml./min. of gas and responds instantaneously to changes in calorific value with an accuracy of within 1·0%.—J. P. S.

High-Velocity Burners Cut Fuel Costs and Furnace Size. L. C. Peskin. (*Iron Age*, 1951, **168**, Aug. 9, 63–66; Aug. 16, 104–107). A new type of burner, for gas or liquid fuels, which may be used in reheating, continuous annealing, heat-treating, and for ore beneficiation and refining plants is described. Mixing and flame-holding conditions are such that the burning can be maintained at extremely rapid rates with a high rate of heat release in excess of 1 million B.Th.U./cu. ft. of combustion space. A non-luminous flame produces clean, soot-free convection heat and non-oxidizing atmospheres can be readily obtained. Details of the design for a steel strip annealing furnace are given.—A. M. F.

Heat Transfer and Turbulence in Gases Flowing Inside Tubes. S. I. Evans and R. J. Sarjant. (*J. Inst. Fuel*, 1951, **24**, Sept., 216–227). This paper describes a series of experimental investigations into the influence of turbulence in heat transfer from gases flowing in tubes, together with an examination of the influence of the separate contribution to the total heat transfer made by convection, as distinct from that made by non-luminous gas radiation.—J. P. S.

Where the Coal Goes. H. Buckley. (*Mech. World*, 1951, **129**, June 15, 555–559). The paper is a concise survey of the background of the fuel situation for the information of the fuel user. Output, consumption, manpower, and wages are discussed with graphical aid.—D. H.

Recent Developments in Coal Preparation. W. R. Chapman. (*J. Inst. Fuel*, 1951, **24**, July, 168–172). Recent developments described include the Humboldt and Vögel cleaning processes, the Dutch State Mines cyclone as a means of replacing thickeners in the water circuit of jig washers and as a means of cleaning slack coal, and a modification to the standard method of operating jig washers introduced by B. M. Bird in the U.S.A. Results from a jig washer on the Bird system are reported.—J. P. S.

Cyclone Type of Washer for Coal Cleaning. A. Lahiri. (*J. Sci. Indust. Res.*, 1951, **10A**, Aug., 317–325). The author critically reviews the literature on the cyclone washer. The design and operation of the cyclone washer are described.

Swelling Power of Coal. C. E. Spooner. (*Fuel*, 1951, **30**, Sept., 193–202). A general relationship is deduced showing that the swelling power of coal is directly proportional to the tar produced on thermal decomposition and inversely proportional to the amount of porous structure of the coal. This has been reduced to the general statement "swelling $\propto$ (hydrogen $-$ 4·2)/(oxygen)^2." This relation is shown to apply

and equations are deduced for swelling tests with fast and slow rates of heating such as the British Standard swelling test, the Gray-King coke type, and the Sheffield Laboratory coking test. These formulæ are then applied to the deduction of the calorific value, ultimate analysis, and other properties of coals.—J. P. S.

Macro-Pore Size Analysis of Metallurgical Coke. J. D. Gilchrist and J. Taylor. (*J. Inst. Fuel*, 1951, **24**, Sept., 207–211). Possible methods of estimating the sizes of pores in coke are reviewed. The only one considered practicable is that based on the supposition that as the particle size diminishes pores are destroyed in order of size and there is consequently a change in apparent density which can be estimated from the average weight per particle. Apparent densities are determined on large pieces and, by a new method, on fine particles; the data are used to calculate apparent densities and then the porosities for the intermediate particle sizes. Finally, the maximum pore size corresponding to a given particle size is determined by microscopic examination. Pore-volume/pore-size distribution curves are plotted, which show that, except in petroleum coke, there are two distinct groups of pores, one of size 0·05 to 0·5 mm. and the other 1·0 to 2·0 mm.—J. P. S.

Coke Screening. A. A. Milkie. (Blast Furnace and Coke Assoc. of the Chicago District: *Blast Furn. Steel Plant*, 1951, **39**, Sept., 1091–1094). 'Flat-top' woven screens give accurate sizing with the least resistance to flow. Screens with square openings give the most efficient and accurate sizing. The tensioning of the screens is discussed and suggestions are given for installation and maintenance.—J. P. S.

Firing with Fuel Oil—Technical Aspects of Combustion of Fuel Oils in Comparison with Other Fuels. B. Bengtsson. (*Blad för Bergshandteringens Vänner*, 1950, **29**, No. 4, 174–193). [In Swedish.] The author compares fuel oils with coal and producer gas for use in the open-hearth process and steam generation. The relative advantages of oil as fuel are chiefly saving of labour, less repairs, cleanliness, and often an increase in production. The high flame radiation occurring in oil firing makes it specially suitable for the open-hearth furnace. Tables of data are presented.—B. S. E.

Firing with Fuel Oil—General Technical and Economic Aspects. J. M. Schenström. (*Blad för Bergshandteringens Vänner*, 1950, **29**, No. 4, 147–173). [In Swedish]. An exposition of world demand and supply of oil is given. Testing standards, transport, combustion economy, and the influence the sulphur in fuel oil has on the open-hearth furnace process are discussed.—B. S. E.

REFRACTORY MATERIALS

Ceramics as Gas-Turbine Blade Materials: A Survey of the Possibilities. T. G. Carruthers and A. L. Roberts. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, pp. 268–273). The potentialities of ceramics for use in gas turbines are reviewed under two headings; (*a*) Multi-component materials forming high-temperature eutectics and (*b*) single-component materials.

The absence of ductility and the sensitivity to thermal shock and inconsistency of properties are seen as their main disadvantages. Possible means of increasing the ductility and resistance to thermal shock are discussed. Available data on the mechanical properties of possible ceramic turbine materials are tabulated. It is concluded that some single-component materials are potentially suitable for use in turbines provided that operating conditions are such as to minimize thermal shock. It is believed that such materials are capable of further development.

Ceramics for Gas Turbines. L. Rotherham, W. Watt, J. P. Roberts, and F. J. Bradshaw. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, pp. 273–280). The preparation of sintered alumina and methods of measuring its mechanical properties are described. The results of bend strength, tensile strength, Young's modulus, and creep determinations are included. The methods used are applicable to ceramic materials in general. Experimental and theoretical approaches to the thermal shock problem of ceramics are also given.

Ladle Refractories and Practice in Acid Electric Steel Foundry. C. H. Wyman. (*Amer. Found. Soc. Preprint No.* 64, 1951). The properties required from ladle refractories are discussed, and suitable mixtures for specific parts of ladles are given. All aspects of ladle bricking, preheating and drying, and stopper-rod preparation are described.—P. C. P.

BLAST-FURNACE PRACTICE AND PRODUCTION OF PIG IRON

Chemistry in Metal Extraction. F. D. Richardson. (Brit. Assoc.: *Engineering*, 1951, **172**, Nov. 2, 569–571). The broad chemical principles of smelting and refining are discussed, the parts played by slags are considered and the chemistry of molten metals is referred to together with the work of Schenck, Chipman, and McCance. The author suggests that models of molten metals and slags should be based on the knowledge of chemical structure; these could be developed in the light of thermodynamic information. These models would indicate how far existing thermodynamic knowledge could safely be extrapolated beyond the range of conditions over which it has been established. Reference is made to the clustering of atoms in certain complex solutions; and the behaviour of oxygen in iron containing chromium and of sulphur in iron containing manganese is given as strong evidence for clustering between pairs of solutes. The effect of such mutual interactions may influence greatly the behaviour of both impurities and alloying elements. The atomic nature of slags is also discussed. Lastly the author shows how research on slags may lead to the recovery of about 100,000 tons of sulphur from the 7,000,000 tons of blast-furnace slag produced annually in Great Britain.—M. D. J. B.

Development of the V.Ö.E.S.T. Blast-Furnace Plant. A. Uxa. (*Vereinigte Österreichische Eisen- und Stahlwerke Jahrbuch* 1950–51, 25–30). The efficiency and the performance of the furnaces, mainly as related to the composition of the burden during the 1939–45 war and post-war years, are analysed. The proportion of sintered ore in the burden increased from about 15% of the iron-bearing charge to about 45% during the war. From 1947 to 1950 the sinter remained at about 65%. Pig-iron production as well as coke consumption and man-hours per ton of pig iron produced are shown for the post-war period, and an analysis of the allocation of the pig iron for steelmaking and for castings is made.—P. F.

Development Trends In Iron and Steel Making. B. Kalling. (*Jernkontorets Annaler*, 1951, **135**, No. 7, 241–286). [In Swedish]. The author gives a broad survey of recent developments in ore beneficiation and reduction with mention of the electric low-shaft furnaces now in operation, pig-iron refining, and the different steel-melting processes.—B. S. E.

Low-Shaft Iron Smelting Furnaces. D. J. O. Brandt. (*Coke and Gas*, 1951, **13**, Oct., 351–356, 364). The ordinary blast-furnace, electric low-shaft furnace, and oxygen-enriched low-shaft furnace are briefly discussed; the briquette-charged low-shaft furnace is also mentioned.—T. E. D.

PRODUCTION OF STEEL

Scrap. (*Usco Magazine*, 1951, **2**, June, 6–8). A brief description is given of the working of the scrapyard at the Vaal Works of the Union Steel Corporation of South Africa.—D. H.

Twenty-five Years of the Klip Works. 4—Amazing Progress in the Last Ten Years. (*Usco Magazine*, 1951, **2**, June, 23–25). The part played by the Klip Works of the Union Steel Corporation of South Africa in the 1939–45 war is described. Details are given of the post-war development plan.—D. H.

The Development of the V.Ö.E.S.T. T. E. Suess. (*Vereinigte Österreichische Eisen- und Stahlwerke Jahrbuch* 1950–51, 8–15). A description of the construction, between 1939 and 1945 of the V.Ö.E.S.T. iron and steel works at Linz, Austria, is given. A plan of the works, and its production during and after the war, when it was rebuilt after almost complete destruction, is presented. Information on fuel and power consumption as well as slag utilization is given.—P. F.

Ford Motor Company Rounds Out Steel Plant Operating Units for Balanced Production. C. Longenecker. (*Blast Furn. Steel Plant*, 1951, **39**, Aug., 926–957). Supply and storage facilities are described. Operational details are given of the coke plant, blast furnaces, electric furnaces, open-hearth furnaces, rolling mills, and power station. Auxiliary equipment in each department is discussed.—J. P. S.

Converter Steel Production with Oxygen Injection. T. E. Suess. (*Vereinigte Österreichische Eisen- und Stahlwerke Jahrbuch* 1950–51, 21–24). Oxygen was blown from the

top into the charges of 2-ton and 15-ton experimental, converter-shaped vessels, and a considerable improvement, particularly a reduction in nitrogen content of the steel, made from Styrian pig iron, was obtained. Experimental details and results are given. A diagram showing the relation of phosphorus in steel to the CaO/SiO_2 ratio in the converter slag is shown.—P. F.

Development of Large Open-Hearth Furnaces. H. Trenkler and F. Klepp. (*Vereinigte Österreichische Eisen- und Stahlwerke Jahrbuch* 1950–51, 31–36). Destruction of blast-furnaces necessitated the use of cold coke-oven gas and oil for firing an existing tilting 200-ton open-hearth furnace. Later a similar furnace was constructed and this was run with blast-furnace gas. A detailed comparison of the performance of the two furnaces is given in tabular form, and improvements in the construction of the second furnace are discussed at length. The burner design is shown.—P. F.

Growth of Cast Irons. Application to the Study of the Growth of Ingot Mould Irons. P. Rocquet and M. Olette. (*Fonderie*, 1951, **67**, July, 2565–2577: *Iron Steel Inst. Translation Series No.* 427, 1951). Determinations of the amount of silicon oxidized, and the microstructures suggest that a growth of 20% by volume occurs in a layer of cast iron up to 10 mm. thick in the middle of the inside faces of ingot mould walls. In the 4-ton mould there is probably a further layer at a depth between 10 and 30 mm. from the inside surface where the growth is 7–8%. The growth is small if the temperature does not reach the transformation point. If the ingot remains only a short time in the mould the temperature may not reach 780° C. and a high-silicon (2%) iron may be used to raise the Ac point. If Ac is bound to be exceeded a low-silicon iron is best.—R. A. R.

Projecting and Depressed Crazing of Ingot Moulds: A Microscopical Examination at the Inner Surfaces of 10-ton Moulds. R. E. Lismer and F. B. Pickering. (*J. Iron Steel Inst.*, 1952, **170**, Mar., 263–267). [This issue].

Insulating Feeder Heads for Steel Ingots and Castings. H. O. Howson. (*Iron Coal Trades Rev.*, 1951, **163**, Nov. 9, 1013–1016). The author discusses the use of insulating materials in feeder heads, and illustrates the greatly improved efficiency of feeding alloy-steel ingots by the use of heads lined with vermiculite slabs or recessed tiles in place of the usual solid tiles or 'compo'-rammed heads. The application of insulating feeder heads to the production of steel castings is then considered, and a number of head designs are suggested, based on the experience with ingot casting.—G. F.

FOUNDRY PRACTICE

The Theory of the Cupola. H. Jungbluth. (*Gjuteriet*, 1951, **41**, Sept., 127–132). [In Swedish]. A generalized formula for the cupola melting capacity as a function of blast volume, coke rate, and combustion rate is attempted. A graphical illustration of the relationships between capacity and iron temperature resulting from the use of preheated or oxygenated blast with varying blast volume and coke rate is given.—B. S. E.

Patching of Cupola Furnaces by Spraying. O. Göransson. (*Gjuteriet*, 1951, **41**, Sept., 133–136). [In Swedish]. Considerable savings of material and labour can be achieved by using a cement spraying gun for the routine repair of cupola linings.—B. S. E.

Some Trends in the Production of Nodular Cast Iron. J. A. Bressel Egido. (*Inst. Hierro Acero*, 1951, **4**, Apr.–June, 112–130; July–Sept., 197–208). [In Spanish]. Mainly American and British methods of making nodular cast iron with magnesium are surveyed. Trials carried out by the author at the Saragossa Works of the Ebro Engineering Foundries are described. Preliminary tests, even with metal containing up to 0·25% S, have indicated the possibility of producing this material in Spain on a commercial scale. Details are given of the charge and method of working, and of forging tests. Structure and mechanical properties are described. A method of determining magnesium in nodular cast iron using 8-hydroxiquinoline is described.—R. S.

Notes on the Formation of Nodular Graphite, in Magnesium-Treated Cast Iron. F. Muñoz del Corral. (*Inst. Hierro Acero*, 1951, **4**, July–Sept., 227–229). [In Spanish]. J. Navarro recently indicated new aspects of the formation of nodular graphite and the major importance of the phenomena in the intermediate phases in the formation of the nodular structure. The present note suggests a mechanism for nodules according to which they germinate in preferred regions of the austenite–cementite boundaries, and grow by precipitation of carbon from the austenite and progressive solution of the cementite in the austenite.—R. S.

Centrispun High Alloy Steel Aero-Engine Components. Part I—The Centrispinning Process. A. E. Thornton and J. I. Morley. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 189–194). Extensive use has been made by the aircraft industry of centrispun die castings of certain highly alloyed heat-resisting steels. This paper outlines the main features of the centrispinning process and discusses its advantages and limitations from a metallurgical viewpoint. Results of mechanical tests on sections cut from representative castings are given. The high order of the tensile strength and ductility obtained confirm the superiority of the centrispun die process over the older methods of gravity casting.

Centrispun High Alloy Steel Aero-Engine Components. Part II—Physical and Mechanical Properties of Centrispun Die Castings. J. I. Morley. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 195–205). This Part contains a detailed statement of the physical and mechanical properties, at both room and elevated temperatures, of typical castings made by the centrispinning process. The steels used contained 18–25% of chromium and 8–14% of nickel, with the addition of a small proportion of one of the elements, titanium, niobium, or tungsten. Attention is given to the microstructures and their correlation with mechanical properties. The sensitivity of one of these steels to changes of chemical composition is the subject of an Appendix.

Centrifugal Steel Castings for Gas Turbines. J. Taylor and D. H. Armitage. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 205–208). The evolution of the production of centrifugal castings is briefly dealt with and the fields of application of the horizontal and vertical axis methods are outlined. A complete description is given of the method of manufacture and of the stringent inspection procedure applied in order to attain high standards which are essential for production of gas-turbine components. The results of mechanical tests taken from the castings themselves substantiate the claim that these are at least equal to those obtained in the highest-grade wrought products.

Investment-Casting of Nozzle Guide Vanes. H. E. Gresham and A. Dunlop. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 209–212). This paper describes in detail the modern development of the old 'lost-wax' process of casting as applied to the manufacture of nozzle guide vanes for gas turbines. Factors affecting dimensional accuracy are discussed. Some of the creep properties of investment-cast alloys suitable for nozzle guide vanes are given.

Precision-Casting of Turbine Blades. E. R. Gadd. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 212–216). Turbine blade castings are being made successfully by the 'lost wax' precision-casting method, and the present paper describes briefly the methods used in a particular foundry. Emphasis is placed on the need for precise control at all stages and particularly in the preparation of the wax model. The casting defects likely to be met with, and their causes, are detailed. Finally, some information is given on the high-temperature fatigue properties of cast alloys and a laboratory method for determining the resistance to thermal shock is outlined.

Concerning Synthetic Moulding Sands. H. Neudecker. (*Vereinigte Österreichische Eisen- und Stahlwerke Jahrbuch* 1950–51, 129–132). The influence of moisture on permeability to gas, the shear strength and compression strength in synthetic mould mixtures consisting of various proportions of Austrian constituents, such as silica sand, alumina or bentonite, and anthracite powder was investigated. Moulding sands of satisfactory quality can be developed from locally available materials.—P. F.

Improvement of Synthetic Clay Sands. P. Nicolas. (*Fonderie*, 1951, **66**, June, 2503–2512). The results of studies aimed at establishing the conditions of preparation or of application of synthetic clay sands are given. The following points are discussed: (1) Influence of the grain form of the sand; behaviour at high temperature of the main colloidal

clays on the French market ; high-temperature properties of synthetic sand ; modification of properties by the addition of mineral blacking ; and use of moisture stabilizers.—R. S.

Guide to the Selection, Construction and Operation of Core-Drying Stoves. (*Fonderie*, 1951, **66**, June, 2521–2535). Factors affecting the choice of stoves include the method of heating and hot-gas circulation. Elements to be considered in the construction of stoves include hearths, combustion chambers, burners, recirculation fans, and insulation. The definition of the optimum thermal cycle, precautions on lighting, temperature control, and heat balances are discussed.

Drying of Foundry Moulds and Cores. G. T. Hampton and W. H. Taylor. (*Indust. Gas*, 1951, **14**, Feb., 136–142). The authors describe the core and mould drying equipment at their particular foundry. The old coal-fired batch furnace is mentioned together with its limitations and a comparison made with the latest type of gas-fired continuous oven. The advantages of gas over coke firing and the relative merits of stove and portable hot-air drying equipment for moulds are discussed and brief details given of torch drying.—D. H.

Recent Advances in Dielectric Core Baking. J. W. Cable. (*Amer. Found. Soc. Preprint No.* 10, 1951). The expansion of dielectric core baking from 1945 is breifly reviewed, and developments in equipment are detailed. Resin core binders are considered with reference to reduction of free formaldehyde leading to a considerable reduction in dermatitis and the fumes produced. The advantages of dielectric core baking are enumerated. The costs of orthodox core sands and resin core mixes are compared. Maintenance, fuel, and energy costs, and space requirements are briefly considered. Blowing directly into core driers is described.—P. C. P.

Application of X-Ray Examination to the Improvement of Foundry Technique. J. Oosting and B. Schuil. (*Metalen*, 1951, **6**, Sept. 30, 341–346, Oct. 15, 359–365). [In Dutch]. The first part deals with atmospheric pressure casting. The second part discusses the possibilities of X-ray investigation methods in the foundry, their advantages over mechanical tests, and their effect on cost. Finally, the field in which radioactive isotopes can be used is indicated.—R. S.

Freezing of White Cast Iron in Green Sand Molds. H. A. Schwartz and W. K. Bock. (*Amer. Found. Soc. Preprint No.* 77, 1951). Data obtained during experiments on the graphitization of white cast iron were used to test Chvorinov's theory that the freezing time is proportional to the square of the ratio of the volume of a casting to its area. It was found, using the normal cooling-curve technique that the freezing time was roughly proportional to the 1·5 power of volume/area.

Solidification of Gray Iron in Sand Molds. R. P. Dunphy and W. S. Pellini. (*Amer. Found. Soc. Preprint No.* 22, 1951). Thermal studies of the solidification of grey irons from sand walls have shown that solidification proceeds in a wave-like fashion by the travel of ' start ' and ' end of freeze ' waves. Austenite dendrite ' start ' and ' end ' waves travel in sequence, *i.e.*, the ' start ' wave completes its travel to the centre of the casting before the ' end ' wave begins, thus creating a semi-solid condition through the entire casting. Eutectic formation proceeds by the travel of ' start ' and ' end ' waves in conjunction ; *i.e.*, as a narrow band. In each case the ' start ' waves move through isothermal liquid. The effects of superheat, carbon equivalent, and sea coal modify the timing and rate of progression of the freezing waves.—P. C. P.

Dimensional Checking and Pressure Testing of Gray Iron Castings. K. M. Smith. (*Amer. Found. Soc. Preprint No.* 14, 1951). The reasons for dimensional checking are first considered, with a description of the various methods used in American foundries. Several illustrations of gauges and jigs are given. The different pressure-testing techniques are described with recommendations on the best methods. Air-tight sealing plugs and pads are illustrated.—P. C. P.

Study on Surface of Casting. VI—Considerations on the Sieving Method of Molding Sand. K. Katori, T. Okakura, and K. Hashimoto. (*J. Mech. Lab., Japan*, 1950, **4**, No. 7, 274–278). [In Japanese]. Sieving is experimentally investigated with reference to sieving time, size of sieve openings, and shape of openings.

Study on Surface of Casting. VIII—Considerations on Measuring Methods of Casting Surface. K. Katori, T. Okakura, and K. Hashimoto. (*J. Mech. Lab., Japan*, 1950, **4**, No. 8, 304–308). [In Japanese]. The light cross-section method and tracer method of examining surface characteristics are discussed.—T. E. D.

HEATING FURNACES AND SOAKING PITS

Automatically Controlled Slot Forge Furnace. (*Indust. Gas*, 1951, **14**, July, 295–302). A description is given of a new slot forge furnace manufactured by the Thermic Equipment and Engineering Co. It is based on reverberatory principles.

Rotating-Hearth Reheating Furnace. (*Iron Coal Trades Rev.*, 1951, **163**, Nov. 9, 1035). An oil-fired rotating-hearth furnace, developed by Stein et Roubaix, for reheating certain steel components which are difficult to reheat in continuous pusher-type furnaces is described. The furnace is divided into zones for preheating, heating, and soaking, each zone being separately controlled ; its capacity is 20 tons/hr.—G. F.

HEAT-TREATMENT AND HEAT-TREATMENT FURNACES

Stanelco Electro-Gas Heating Equipment. (*Mech. World*, 1951, **130**, July 6, 9–10). A description is given of new electro-gas equipment recently added to their range by Standard Telephones and Cables, Ltd. The apparatus provides quick local heating for soldering, brazing, annealing, and hardening, and combines the advantages of the flexibility and low cost of gas with the exact control and precision of electrical apparatus.—D. H.

Furnace Efficiency : Improved Designs for Heat Treatment and Mould Drying. (*Iron Steel*, 1951, **24**, Oct., 487–490). The importance of ensuring uniformity of hearth temperature, and full utilization of the heat input, in the design of iron and steel auxiliary furnaces is discussed with special reference to mould drying stoves designed by John Mathison, Ltd., and to oil-fired and gas-fired heat-treatment furnaces, in which the avoidance of direct flame contact with the stock is important. Data on the performance of these furnaces are given.—G. F.

Machine Cuts Heat Treat Furnace Loading Costs. W. G. Patton. (*Iron Age*, 1951, **168**, Aug. 9, 68–70). A new loading and metering device consists of vertically moving, cam-actuated pushers separated by fixed spacers. These pushers lift material from a hopper and discharge it in even quantities to a conveyor belt feeding a continuous heat-treatment furnace.

To Save Money, Conserve Alloy, Repin Your Furnace Belts. R. Williams. (*Iron Age*, 1951, **168**, Aug. 16, 102–103). Experiences with the repair of alloy link conveyor belts used in cyaniding furnaces are described. Failure usually starts with the link pins which can fairly readily be replaced. Other considerations, such as relieving the tension when the belt is not in use and reoxidation of the alloy parts by admitting air occasionally into the furnace, will also help to increase belt life.—A. M. F.

The Importance of the TTT-Curve to the Steel User. R. Mitsche and A. Legat. (*Betrieb u. Fertigung*, 1951, **5**, June, 93–94). The derivation and meaning of the time–temperature–transformation (TTT) curve for isothermal transformations are discussed. Such curves can only be directly applied by the steel user for certain limited purposes. It must be supplemented by cooling-transformation curves, which can be directly applied to continuous cooling processes.—H. R. M.

The Heat Treatment of Bearing Races. (*Indust. Gas*, 1951, **14**, May, 225–229). Details are given of the construction and operation of a gas-fired, pit-type, gas-carburizing furnace designed by Birlec Ltd. and installed at the Aston Works of British Timken Ltd. for carburizing outer and inner rings for heavy industrial tapered roller bearings. It employs U-shaped recuperative radiant tubes as the heating elements.

The Nitriding of Special Steels. J. Daurat. (*Mét. Constr. Mécan.*, 1951, **83**, Apr., 255–258, 266 ; May, 363–371 ; June, 479–483). The part played by nitrogen in steelmaking is briefly considered and the principles of the nitriding process are described. The effects of alloying elements on the hardness obtained by nitriding are discussed. The surface and depth of hardness of several steels are considered in relation to the nitriding treatment. Several Nitralloys, including graphitic and austenitic types, are described. The economic aspect of the nitriding of stainless steels is considered. Methods of protecting surfaces not required to be nitrided are explained.—R. S.

FORGING, STAMPING, DRAWING, AND PRESSING

Stainless Jet Engine Stampings Mass-Fabricated on Production Line. G. C. Close. (*Steel*, 1951, **129**, Oct. 29, 64–65).

At the Solar Aircraft Co., San Diego, California, fully mechanized production lines for precision stainless-steel inner and outer combustion chambers are being completed. The author describes how mechanization is responsible for the high production rates and careful inspection for the remarkable precision achieved.—M. D. J. B.

Profile Measurement of Wire-Drawing Dies: Recent German Developments. J. G. Wistreich. (*Iron Coal Trades Rev.*, 1951, **163**, Nov. 2, 965–968). The shape of the die used in wire-drawing operations is a critical factor governing the efficiency and uniformity of deformation of the wire. The author describes two instruments, the Alfameter and the Zetmeter, which have been developed at the Max Planck Institut, Düsseldorf, for the rapid checking of die profiles. It is suggested that the use of these instruments, in conjunction with a precision measuring instrument such as the Brit. Iron Steel Res. Assoc. profilometer, will enhance the precision of die profiles and enable the best profile to be chosen for a given purpose.—G. F.

ROLLING-MILL PRACTICE

Maintenance for 600 Series Auxiliary Mill Motors. J. C. Anderson. (*Blast Furn. Steel Plant*, 1951, **39**, Sept., 1077–1083). The author discusses the standardization of auxiliary mill motors. The maintenance of the commutator, brushes, brush holders, and bearings is outlined.—J. P. S.

On the Rolling Friction. T. Hisaka and K. Tsugawa. (*J. Mech. Lab., Japan*, 1950, **4**, Mar., 9–21). [In English]. The gravitational pendulum method and the elastic pendulum method, the relations between rolling friction and load, radius of curvature, contact length, and material and surface condition are investigated. Particular reference is made to the friction of knife edges.—T. E. D.

The New V.Ö.E.S.T. Sheet Bar Mill. H. P. Weitzer. (*Vereinigte Österreichische Eisen- und Stahlwerke Jahrbuch* 1950–51, 16–20). A description and engineering drawings of the semi-continuous strip mill at the V.Ö.E.S.T. works, Linz, Austria, are given.—P. F.

Cold Rolling of Steel Strip. C. Sebardt. (*Jernkontorets Ann.*, 1951, **135**, No. 7, 287–402). [In Swedish]. Both the theory and practice of cold rolling are dealt with in this comprehensive paper. Different methods of calculating roll forces and several other factors are explained. Material properties in relation to degree of reduction and number of passes, tolerances, and surface defects are discussed. Descriptions are given of all processes connected with cold rolling, and of rolling mills, furnaces, and equipment used in pickling, annealing, rolling, shearing, and inspection. The economics of cold rolling in Sweden are considered.—R. A. R.

Columbia's Cold Reduced Steel Products. L. S. Dahl. (Amer. Inst. Min. Met. Eng.: *Blast Furn. Steel Plant*, 1951, **39**, Sept., 1084–1090). The sheet and tinplate mill and annealing plant at the Columbia Steel Company's works, Pittsburg, California, are described. In the 5-stand 4-high cold-reducing mill steel is reduced to 0·0035 in. thick at a finishing speed of 3900 ft./min. The annealing department comprises ten gas-fired radiant-tube rectangular furnaces which can be raised, moved, and lowered over cylindrical covers supplied with a protective atmosphere in which the coils are stacked.

Faster Mill Speeds Increase Rolling Capacity. D. Reebel. (*Steel*, 1951, **129**, Oct. 15, 88–98). The author shows how rolling-mill delivery speeds have increased from 25% to 40% during the last few years. Mill engineers are now adopting sealed mercury-arc rectifiers for supplying direct current for many of their electrolytic processes.—M. D. J. B.

A Strip Mill Flying Shear. (*Canad. Metals*, 1951, **14**, July, 10–11, 14). An electric control system for a flying shear, designed by the English Electric Co. of Canada, uses a 'magnavolt' speed control with superimposed electronic trimming. Acceleration control, also electronically trimmed, ensures correct shear speed when the knives meet. All variations of conditions are allowed for and lengths of plate are cut consistently to within 1 in. of the desired length. This applies over the whole range of operating conditions, *i.e.*, 800 to 1400 ft./min. strip speed and 12 to 22 ft. cut length.—E. C.

Consumer Installs Slitting and Leveling Plant. (*Iron Age*, 1951, **168**, Aug. 23, 78–80). Flexibility and greater economy have been obtained with new slitting, shearing, and levelling equipment at the works of E. F. Hauserman Co., Cleveland. Equipment includes an uncoiler, gang slitter, Hallden flying shear, Voss roller-leveller, runout table, oiler, piler, conveyors, and baller to process waste material.—A. M. F.

Mechanical Aids in the Operation of Rolling Mills. J. S. Mayne. (*J. Jun. Inst. Eng.*, 1951, **61**, July, 337–356). The author gives a brief survey of mechanical aids used in the operation of rolling mills. 'Mechanical aid' means apparatus necessary to perform the hot and heavy operations which, except for temperature and bulk considerations, would be performed by hand. Several examples are described.—D. H.

MACHINERY FOR IRON AND STEEL PLANT

Margam Steelworks. (*Electrician*, 1951, **147**, July 27, 253–260). The main electrical features of the Margam-Abbey Works of The Steel Co. of Wales are described. Reference is also made to the partially completed tinplate factory at Llanelly.—D. H.

Overhead Handling Facilitates Automobile Seat Spring Production. F. C. Badke. (*Steel*, 1951, **129**, Oct. 29, 70–71). This article discusses the advantages of overhead handling equipment and describes the Bedford, Ohio, plant of the Universal Wire Spring Co. which manufactures corrugated springs for automobile seats. From wire storage to the dispatch department all handling is done by overhead tram-rail conveyors with rack carriers.—M. D. J. B.

Material Handling for a Modern Open Hearth Furnace Plant. D. J. Loughrey. (Amer. Soc. Mech. Eng.: *Blast Furn. Steel Plant*, 1951, **39**, Oct., 1211–1215, 1272–1273). Material handling with reference to new installations at the Pittsburgh works of the Jones and Laughlin Steel Corp. is discussed. The new unit will contain 11 basic open-hearth furnaces, with a rated capacity of 250 tons each.—J. P. S.

Steelworks Cranes. I. M. Slade. (*Mech. Handling*, 1951, **38**, Oct., 385–388). The paper is a summary of the improvements in and the developments made possible by four years of collaborative research between Brit. Iron Steel Res. Assoc., steelmakers, and crane builders. The author discusses briefly the new Brit. Iron Steel Res. Assoc. specification for steelworks cranes.—D. H.

Materials and Performance. A. T. Bowden and W. Hryniszak. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 11–17). An assessment is made of some of the effects on the performance of the industrial gas turbine resulting from the use of high-alloy steels. In terms of these factors consideration is given to the effect of using such materials upon the construction of the most essential components of the turbine, *viz.*, combustion chamber and heat-exchanger, partly from experience gained with the Parsons experimental gas turbine. The influence of the material on the construction of the compressor is also dealt with. The choice of material is governed by the gas temperature, which is dependent on the required turbine inlet temperature. The effect of the turbine inlet temperature on the performance of the gas turbine is studied within the general framework of the paper. The effect of artificial cooling is also briefly considered.

Influence of Operating Temperature on the Design and Performance of Gas Turbines. S. L. Bragg. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 23–29). The primary variables in the simple gas-turbine cycle are the maximum (turbine inlet) gas temperature, and the compressor delivery pressure, the component efficiencies being secondary variables: the results in which the user is interested are the specific output which determines the size of the plant, and the overall efficiency, which affects the running costs. Further variables are introduced by the addition to the cycle of other components, such as heat exchangers, intercoolers, and reheat chambers. The relative effects of all these variables are discussed in the first part of the paper, and it is shown that increasing the maximum operating temperature is the simplest method of obtaining worthwhile increases in both output and efficiency.

The second part of the paper deals with the turbine in detail, showing where the physical properties of available materials limit the design. It is suggested that in developing the materials for turbine blading, the emphasis should be on obtaining the same strength at higher temperatures while turbine disc materials will require higher strength at the comparatively low hub temperature, without sacrificing their resistance to oxidation at the hotter rim.

The Effect of Macro-Roughness on the Performance of Parallel Thrust Bearings. M. E. Salama. (*Proc. Inst. Mech. Eng.*, 1950, **163**, 149–161). The problem is treated theoretically and experimentally. Macro-roughness is a prime factor in the behaviour of a bearing as it provides passages which feed the surfaces with lubricant and allows the formation of hydrodynamic films.—R. A. R.

WELDING AND FLAME-CUTTING

Testing of the Susceptibility of Weldable Structural Steels to Cleavage Fracture. E. Folkhard. (*Vereinigte Österreichische Eisen- und Stahlwerke Jahrbuch* 1950–51, 72–81). The determination of the susceptibility of welded structural steels to brittle fracture should be carried out under conditions closely resembling those encountered in actual use. Cleavage fractures are conditioned by triaxial residual stresses in welds; tears are associated with hardening after welding. The notch-impact and Schnadt tests take the effects of specimen dimensions and quality of the material only partly into account and are regarded as unsuitable for the determination of the susceptibility to brittle fracture of welded steels. The Kinzel and bend tests are suitable, provided the testing temperature in the former is lower by 10° C. compared with the latter if comparable results are to be obtained. Full experimental details and photographs of fractures are given.—P. F.

Welding Backing Plates Made of Iron Powder. H. Hauttmann. (*Vereinigte Österreichische Eisen- und Stahlwerke Jahrbuch* 1950–51, 101–103). For eliminating pores in welds and for backing parts such as are necessary to prevent the dripping out of metal when welding **V**-joints, flat sintered rods of iron powder or powdered iron pressed into thin sheet-metal troughs were used. The advantages of this method over those employing plain steel or copper strips are discussed and corroborated by photographs of welds and sections of welds made by the aid of iron-powder fillers in a mild steel and a 0·2% C, 0·3% Si steel.—P. F.

Experiments on the Weldability of Flakes in Chromium–Nickel–Molybdenum–Steels. R. Cabezudo Sanchez. (*Inst. Hierro Acero*, 1951, **4**, July–Sept., 189–196). [In Spanish]. Experiments to demonstrate the weldability of hair-line cracks by forging were carried out at the Reinosa naval works in Spain. A 70% reduction eliminated flakes from a hollow forging even with air cooling without any precautions. Hair-line cracks were deliberately produced in a solid forging which was given an 84% reduction followed by adequate heat-treatment and cooling. No evidence of flakes remained.

R. S.

Welding of Heat Resistant Alloys in Sheet Form. H. E. Lardge. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 217–224). This paper deals with some of the problems which had to be overcome in the development of sheet metal parts for gas turbines and describes some of the applications of the welding processes to the assemblies concerned. A brief description of the sheet metal parts of a typical jet propulsion unit is given first, followed by some notes on the characteristics of the austenitic steels and high nickel chromium alloys which intimately affect welding. Some aspects of the use of oxy-acetylene torch and the carbon, argon, and metallic arc processes are then discussed followed by a description of the techniques developed for the resistance processes of spot, stitch, and seam welding as applied to gas-turbine work. A description of the electrical resistance hot riveting process as developed for use with stainless steel rivets is also given.

Weld-Metal Properties and Welding Characteristics of Two Austenitic Steels Used for Gas-Turbine Rotors. E. Bishop and W. H. Bailey. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 225–232). The weldability, weld metal characteristics, and properties of welded joints have been investigated for two well-known austenitic creep-resisting steels, Jessop *G18B* and *R20*. For each steel, a suitable arc-welding electrode has been developed, capable of depositing weld metal of similar chemical composition to the parent steels. The room-temperature mechanical properties of all weld-metal specimens were investigated and numerous butt joints welded in 1-in. thick plates of the corresponding steels using **U** and **V** preparations. These welds were radiographed and transverse sections cut for mechanical testing. The mechanical properties of the welded joints 'as-welded' and after heat-treatment were investigated at room temperature, and the creep properties at appropriate high temperatures. *G18B* was found to have excellent welding properties, and the welded butt joints were slightly superior to the wrought material in creep properties. With *R20*, cracking trouble was at first experienced due to the formation of an undesirable intergranular brittle constituent in the weld-metal microstructure. Electrodes have been developed, however, which give no cracking trouble. Welded butt joints made with such electrodes were again found to be slightly superior to the wrought steel.

The Welding of Clad Steels. H. B. Pastoor. (*Lastechn.*, 1951, **17**, Oct., 145–148). [In Dutch]. The method of manufacture of clad steel is described. Precautions to be taken in oxy-acetylene cutting of clad steel are given and methods of butt-welding for various types of cladding and thickness are indicated. The welding of flanges and tubing is discussed.

Machining Austenitic and Ferritic Gas-Turbine Alloys. K. J. B. Wolfe and P. Spear. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 233–242). A survey has been made of the problems associated with the machining of heat-resisting and creep-resisting steels and alloys. The results of the work of the B.S.A. Group Machinability Research Laboratory on these types of material covering turning, milling, drilling, broaching, grinding, and examination of cutting fluids are included. Emphasis has been given to the underlying principles involved in the correct selection of conditions for maximum machining efficiency. The survey also includes some information on recent work carried out in the United States and Russia, and lists interesting phenomena encountered in production machining techniques. The paper should assist designers, planning engineers, and workshop personnel.

Sweat-Cooling: A Review of Present Knowledge and Its Application to the Gas Turbine. P. Grootenhuis and N. P. W. Moore. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 281–288). In the method of sweat-cooling the cooling fluid is forced through the pores of the component towards the surface exposed to the heat source. The thermodynamic process is briefly discussed and the relevant literature is reviewed. The flow of fluids through porous materials is considered in some detail. The main object of the paper is, however, to define the material properties required with sweat-cooling and to review the present knowledge on available porous materials. The need for further research is also indicated. It is shown that the sweat-cooling of low-stressed components can be seriously contemplated with present-day materials. Some suggestions are made for reinforcing porous materials. Some tentative designs for gas-turbine blades are also given, including proposals to make a solid core carry the principal loads.

CLEANING AND PICKLING

Some Factors Affecting the Rate of Diffusion of Hydrogen through Steel during Electrolytic Pickling. W. A. Bell, G. J. Metcalfe, and A. H. Sully. (*J. Electrodep. Tech. Soc.*, 1951, **27**, Advance Copy No. 2). Measurements have been made of the rates of diffusion of hydrogen through cylinders of mild and DTD4A steel during cathodic treatment in H_2SO_4 and NaOH electrolytes. The apparent hydrogen diffusion rate varies over wide limits between different electrolytes. With mild steel, increasing cold work increases the rates of diffusion when using H_2SO_4 but with NaOH the reverse seems to hold, except that large amounts of cold work still induce high diffusion rates. The variations are ascribed to the formation of cathodic films which affect the rate of transfer of hydrogen atoms into the metal lattice. The quantity of hydrogen required to cause embrittlement is very much less than that necessary to saturate the steel.—J. P.

The Treatment of Waste Pickle Liquor. (*Indust. Finishing*, 1951, **3**, Apr., 732–733). The liquor is aerated in a storage tank to oxidize all ferrous iron to the ferric condition and caustic soda is added to neutralize all acids. The *p*H of the neutralized waste is measured by an electrode assembly, and

its value is recorded by a potentiometer which automatically controls the supply of caustic soda. Excessive waste of caustic soda is prevented by accurate proportioning to suit the acidity of the waste—E. T. L.

A Method for Disposal of Acid and Alkaline Plating Room Wastes. J. H. Monaweck and C. Kelly. (*Products Finishing*, 1951, **15**, June, 38, 40). Acid and alkaline wastes should be mixed to bring the resultant *p*H to between 4·8 and 9·2 before dumping into sewers.—J. P.

Electrolytic Removal of Galvanic Deposits. A Pollack. (*Metalloberfläche*, 1951, **B**, **3**, Jan., B8–B9). Methods most suitable for the electrolytic removal of nickel, chromium, copper, brass, silver, and gold deposits from metal surfaces are described. Details of the composition of the solutions, of current densities, voltages, and optimum working temperatures are given.—P. F.

German Methods for the Recovery of Zinc from Galvanized Scrap. E. R. Thews and M. Strohmeyer. (*Metalloberfläche*, 1950, A, **4**, Aug., A121–A123; Nov., A169–A172; *Electroplating*, 1951, **4**, Apr., 133–134). Removal of zinc from galvanized articles by volatilization in a rotary furnace or by the action of chlorine is briefly described.—J. P.

Coated Abrasives in the Grinding and Polishing of Stainless Steel. A. E. Brown. (*Electropolating*, 1951, **4**, Mar., 77–80; Apr., 123–126). Machines employing coated abrasive belts for grinding and polishing semi-finished stainless-steel sheets, strip, bar, and tube, and their application are described. Finishing of stainless steel after fabrication usually involves repolishing areas affected during fabrication, such as welds and tool and machine marks, so as to match the finish of the untouched parts. The methods of doing this with various finishes and contours of articles are discussed.—J. P.

Standardization and Simplification in the Coated Abrasives Industry. H. R. Power. (*Products Finishing*, 1951, **15**, June, 18–30). The methods employed to improve the production and usefulness of coated abrasives are described. These include standardization of the abrasive grains with respect to size, physical properties, and cutting power, and improvement of older and development of newer adhesives, backing materials, and methods of coating.—J. P.

Dry Cleaning with Corn Cob Grits. E. W. Shoff. (*Iron Steel Eng.*, 1951, **28**, Sept., 126–128). This article describes a method used for cleaning electrical and other equipment with a stream of air at high velocity containing an abrasive—in this case corn cob grits. It is claimed that corn cob grits yield a soft pellet which has fine cleaning properties, entailing a lesser degree of fire hazard than petroleum solvents; they leave no toxic effects and are non-corrosive. The machinery and operating conditions are described.—M. D. J. B.

PROTECTIVE COATINGS

Studies in the Discontinuities of Electrodeposited Metallic Coatings. II. S. C. Shome and U. R. Evans. (*J. Electrodep. Tech. Soc.*, 1951, **27**, Advance Copy No. 1). A method is described for measuring the total pore area in an electrodeposit of nickel on steel.—J. P.

Review of Finishing Problems in Powdered Metal Products. (*Product Finishing*, 1951, **4**, May, 55–64). The difficulties associated with the preparation and electroplating of sintered, and thus porous, metal components are discussed and suitable procedures recommended. Chemical colouring and other finishes are mentioned briefly.—J. P.

Measuring Adhesion of Electrodeposits. (*Steel*, 1951, **128**, June 11, 107–108). A description is given of a simple and inexpensive method of determining the adhesion of electrodeposited coatings recently developed by the National Bureau of Standards. The technique involves electrodeposition of an adherent, mushroom-shaped nodule on the surface of the coating to act as a grip for the application of a detaching force. The force required to pull off the nodule—together with a portion of the coating—is then obtained on a spring balance—M. D. J. B.

Spiral Contractometer Measures Stress in Electrodeposits. (*Indust. Finishing*, 1951, **3**, Apr., 726–730). The instrument consists of a flat metal strip wound in the form of a helix and connected to a pointer moving over a dial to show the rotation of the free end of the spiral. A single helix can be used almost indefinitely without appreciable change in its dimensions, elastic moduli, or deflection constants. The instrument is easily calibrated.—E. T. T.

Zinc Plating and Lacquer Provide Finish for Steel Conduit. (*Products Finishing*, 1951, **15**, June, 88–90). Electrical conduit made by resistance welding of cold-rolled strip is pickled in sulphuric acid, zinc-plated externally, sprayed with lacquer internally, and dried by hot air fans.—J. P.

Hot Dip Galvanizing of General Work. (*Anglo-American Council on Productivity*, Aug., 1951). This 57-page report presents the observations and conclusions of a specialist team representing the British hot dip galvanizing industry which visited many galvanizing works in the U.S.A. in 1950.—R. A. R.

Hot-Dip Galvanizing of Cold-Rolled Strip. H. Bablik, F. Götzl, and R. Kukaczka. (*Metal Ind.*, 1951, **79**, Sept. 21, 241–243). The surface layer of cold-rolled strip is structurally different from the base metal, and trouble-free galvanizing can only be achieved by first removing this disturbed layer. Both pickling and electrolytic methods are briefly discussed. The authors' own work indicates that anodic corrosion is the most efficient preparation before galvanizing.—P. M. C.

Contact Tinplating with a Difference. J. K. Wilson and O. Wright. (*Electroplating*, 1951, **4**, Sept., 274–276). Exceptionally dense and adherent tin coatings can be formed on most metals, including cast iron, brass, and copper, by simple immersion in contact with aluminium in hot caustic-soda/sodium-stannate solutions normally used for electrotinning. Deposition does not cease when the base metal is covered and fairly thick deposits can be built up in reasonable time. The process, which is equally suitable for large and small parts, is capable of tinning awkward shapes and recesses that cannot be easily coated by electrodeposition; electrotinning of exposed surfaces and contact tinning of recesses can proceed simultaneously. Contact tinning may also be of value as an undercoat in electroplating of cast iron, in the prevention of fretting corrosion, and for the protection of springs and of aircraft radiators, and oil coolers.—J. P.

Co-Deposited Stopoff Conserves Tin. E. S. Coe. (*Iron Age*, 1951, **167**, June 14, 86–88). A co-deposited alloy plating of 5–15% Sn and 85–95% Cu has been found to be an effective substitute for pure tinplating used to form a protective barrier against the furnace atmosphere in selective hardening of parts. An advantage, apart from the economy, is the higher melting point of the alloy and thus less tendency to run in the furnace.—A. M. F.

Tin-Nickel Alloy in Use. S. C. Britton and R. M. Angles. (Electrodep. Tech. Soc.: *Indust. Finishing*, 1951, **3**, July, 966). The authors discuss the corrosion resistance of the new tin-nickel alloy electrodeposit which has been developed in the laboratories of the Institute by N. Parkinson.—E. T. T.

The Development, Production and Manufacture of Electro-Tinplate. W. E. Hoare. (*J. Inst. Prod. Eng.*, 1951, **30**, Mar., 104–133). The cold-rolling process and the shortage of tin have increased interest in electrotinplate. Plating electrolytes are compared and their conditions of use discussed. Ferrostan and alkaline electrotinplate lines and the new halogen line at Weirton, U.S.A., are described.—J. B. H.

Phosphatizing of Steel Components for Corrosion Protection. (*Metalloberfläche*, 1950, A, **4**, Sept., A137–A140). Detailed proposals, co-operatively developed, for phosphatizing steel components are embodied in a draft submitted as a basis for the formulation of German standard specifications.—P. F.

Armed Forces Adopt New Waterproof Packaging. (*Iron Age*, 1951, **168**, July 12, 96–97). A new wrapping for product preservation is composed of layers of cotton scrim, polythene, aluminium foil, and an outer vinyl film.—A. M. F.

Modern Developments in the Effective Utilization of Organic Finishes. H. J. Testro. (*Sheet Metal Ind.*, 1951, **28**, Oct., 955–960, 962). The author reviews traditional and novel methods of applying organic finishes, from the point of view of effective utilization and conservation of costly finishing materials. Consideration is given to flow coating, roller coating, tumbling, whirling, and silk screening; spray finishing techniques including the use of superheated steam, heated paints and lacquers, and electrostatic fields to assist spray deposition are also discussed.—P. M. C.

Metal Spraying in France. J. Cauchetier. (*Electroplating*, 1951, **4**, May, 163–164). The properties of sprayed metal are reviewed. Apart from the spraying of zinc and aluminium on steel for resistance to corrosion and scaling, of lead for resistance to acid attack and for X-ray shielding, and some decorative applications, the most spectacular results have

been achieved in the field of reclamation of worn parts. Automobile and diesel engine crankshafts have been built up by spraying with 1·4% carbon steel and diesel engine cylinder bores have been repaired by depositing up to ½ in. of steel. Other reclamation work included stern post and rudder bearings, railway locomotive induction motor shafts, roll necks, and press tools for motor car bodies.—J. P.

Progress in Metal Spraying Equipment. H. J. Plaster. (*Electroplating*, 1951, **4**, July, 215–217). The history of wire-fed metal spraying is briefly reviewed and some recent developments for automatic gritblasting and spraying of structural steelwork and steel tubing are described.—J. P.

Recent Developments in Metal Spraying by the Powder Process. W. G. McDermott. (*Electroplating*, 1951, **4**, Apr., 114–116, 131). Recent developments in this field are reviewed. The latest Schori pistol produces denser, more adherent coatings of lower oxide content at higher speeds. Efficiency of deposition is increased and application costs lowered. The new pistol is capable of handling metals of high melting point which could not previously be sprayed by the powder process.—J. P.

Some Aspects of Metal Spraying by the Powder Process. J. N. Blake. (*IVA*, 1950, **21**, 1, 27–35). [In English]. Applications of the Schori spraying process using metal powders or thermoplastic polymers sprayed by compressed air from a pistol, and melted by various gas mixtures are exemplified. Details of the spray gun are given.—B. S. E.

Automatic Metal Spraying of Steel Tubes. (*Indust. Finishing*, 1951, **3**, Apr., 712–716). The paper describes the automatic aluminium-spraying plant operating at the works of Scaffolding (Great Britain) Ltd. The tubes sprayed are up to 20 ft. long and 2 in. in dia.—E. T. T.

Sprayed Metal Coatings on Polished Surfaces. R. Corbett. (*Indust. Finishing*, 1951, **3**, July, 955–961). **Molybdenum in Metal Spraying by the Wire Process.** R. Corbett. (*Electroplating*, 1951, **4**, Feb., 45–48, Mar., 84–88). This article describes and illustrates the uses of molybdenum metal in surface preparation. When spraying molybdenum it is possible to deposit a clean metal free from oxide on the polished metal with adhesion of the order of ½ ton/sq. in. An oxy-acetylene flame gives the best results. Acetylene/oxygen ratios are critical. Micrographs of the molybdenum–steel interface show a very close physical contact between the two metals, but no alloying is revealed. On further heat-treatment at 1000° C. in hydrogen, the molybdenum coating adheres firmly because alloying takes place. Heat-treatment in nitrogen renders the coating brittle.—E. T. T.

Problems in Metal-Spraying Technique. Püschel. (*Metalloberfläche*, 1950, A, **4**, Sept., A141–A143). The scope of metal-spraying techniques in their present stage of development is assessed. The various types of fixed and transportable spraying equipment are considered, the methods of gas mixing, the use of town gas, optimum dimensions of the metal wire, hardness of the sprayed deposits, the use of nitrogen in place of compressed air, repair of worn parts by spraying, and other questions are discussed.—P. F.

On Accident Prevention in Flame-Spraying of Steel, Metals and Plastics. I. C. Fritz. (*Metalloberfläche*, 1951, B, **3**, Jan., B7–B8). Possible causes of accidents at works using metal-spraying guns are examined, and the appropriate safety measures in particular applications are discussed.—P. F.

Metal Surfacing by the Spraying Method of J. Cauchetier. H. Reininger. (*Metalloberfläche*, 1950, **4**, Aug., A119–A121). Cauchetier's research on spray-gun metallizing is discussed with special reference to German problems. The structures and compositions of sprayed-on surfaces are examined, and the application of the method to the repair of worn car cylinders, the production of lubricated surfaces and the resurfacing of worn crankshafts are mentioned as examples of its economical and versatile use.—P. F.

Deposition of Iron on Ceramic Components in a Protective-Atmosphere Electric Furnace. K. Backhaus. (*Elektrowärme-Tech.*, 1951, **2**, Aug., 80–84). To facilitate hard-soldering of metal components to ceramic insulating materials, metallizing of the ceramic surfaces is necessary. Three processes by which a suspension of iron in oil is applied to the ceramics and subsequently burnt on to the surface in a reducing atmosphere are described. The electric furnaces employed and their construction are discussed.—P. F.

Heat-Resistant Ceramic Coatings Broaden Low Carbon Steel Applications. (*Steel*, 1951, **129**, Aug. 13, 74–77, 96, 100). Details are given of heat-resistant ceramic coatings developed for use in a wide range of temperatures and on a variety of metal surfaces. The importance of base-metal preparation is stressed, and firing temperatures are considered together with coating thicknesses. The practical applications of ceramic coatings on steel are discussed. Properly applied coatings will increase the fatigue strength and fatigue life of parts.—M. D. J. B.

Metallurgical Aspects of the Vitreous Enamelling of Cast Iron. A. L. Taylor. (Australian Inst. Met.: *Australasian Eng.*, 1951, June 7, 73–81; July 7, 69–73). The author first considers in detail the design, composition, and surface preparation of castings suitable for vitreous enamelling, and then discusses foundry and moulding practice. Frit making is next dealt with; compositions, smelting, and milling practice being described in detail. Various methods of enamel application and firing, and the many defects which may spoil the finished coat are described. (96 references.)—P. M. C.

Gas Evolution from Gray Cast Iron During Enamelling. L. F. Porter and P. C. Rosenthal. (*Amer. Soc. Met. Preprint* 34, 1951). Gassing is due mainly to the CO and CO_2 from the iron surface, evolved soon after the enamel is molten. Hydrogen is evolved while the enamelled plate is cooling, and causes blisters.—E. T. L.

Infra-Red Lamps Speed Drying in Pacific Coast's Largest Auto Paint Shop. (*Products Finishing*, 1951, **15**, June, 92). The methods by which motor-car bodies are cleaned, sprayed with synthetic enamel, and dried by infra-red lamps are described.—J. P.

Blitz Cans Receive Corrosion Resisting Specification Finish in Production Finishing Set Up. E. A. Blount. (*Products Finishing*, 1951, **15**, July, 38–52). The production and painting of petrol containers ('Jerricans') are described.

Aerocoach Bus Finishing Aided by Natural Gas. A. Q. Smith. (*Products Finishing*, 1951, **15**, Aug., 26–32). The construction and painting of passenger motor coaches are described. Natural gas is employed for heating cleaning tanks and for paint-drying and stoving ovens.—J. P.

A Study on the Electrostatic Spray-Coating Process. Part I. S. Yoshida, S. Okamoto, and K. Tamura. (*J. Mech. Lab.*, 1950, **4**, No. 6, 224–231). [In Japanese]. The fundamental conditions for electrostatic spray-coating were investigated experimentally. In addition, quantitative tests showed, by employing suitable conditions, that the efficiency of spraying could be raised from 20–30% to 50–60% using an electrostatic field. From further experimental work, the mean magnitude of electric charge on a paint particle was estimated.—T. E. D.

Improved Enamels Extend Life of Steel Chlorination Pipe. L. E. Dube and N. Stromdahl. (*Steel*, 1951, **129**, Oct. 22, 70–72, 99). The development of a protective ceramic which would lengthen the life of black steel chlorination tubes is described.—M. D. J. B.

The Elements of the Third, Fourth, and Fifth Series as Possible Adherence-Promoting Materials for Sheet-Iron Enamels. J. H. Healy and A. I. Andrews. (*J. Amer. Ceram. Soc.*, 1951, **34**, July, 214–219). The properties of the elements of the third, fourth, and fifth series of the Periodic System are analysed with regard to their adherence-promoting qualities.—E. C.

The Cobalt-Reduction Theory for the Adherence of Sheet-Iron Ground Coats. J. H. Healy and A. I. Andrews. (*J. Amer. Ceram. Soc.*, 1951, **34**, July, 207–214). New data concerning the adherence phenomenon of cobalt ground-coat enamels were obtained by microscopic observation during and after firing, and by X-ray spectrometry using an iron target. Details are given of techniques and results.—E. C.

Ceramic-Coated Exhaust Systems. W. G. Hubell. (*Aircraft Production*, 1951, **13**, Nov., 357–362). Several aircraft exhaust-system components were manufactured by the Ryan Aeronautical Company, California, of ceramic-coated 19/9 DL stainless steel, uncoated 19/9 DL, and a variety of other uncoated alloys. These components were then used on the engines of Boeing 377 Stratocruiser aircraft engaged in trans-Pacific flights. After 650 hr. service, removal and examination of these parts showed that the ceramic coatings on the 19/9 DL test parts had completely protected them from deterioration through oxidation, carbon absorption, and corrosion attack under operating temperatures up to 1800° F. The coatings, which are from 0·001 to 0·002 in. thick, are

capable of withstanding fairly rough handling, and are not affected by thermal shock at any temperature between −75° F. and 1700° F.—T. E. D.

Productivity Report on Metal Finishing. (*Anglo-American Council on Productivity*, 1951). This is the report of a team which visited the U.S.A. in 1950 to investigate two main classes of metal-finishing process, namely, painting and electroplating, as carried out in America. The recommendations include: (1) Management should make available as many cost data as possible to the whole staff; (2) trade associations should educate both industry and the public to appreciate the significance of finish and finishing operations; (3) there should be much closer collaboration between design and finishing departments; (4) greater attention should be paid to process and quality control; (5) new methods of paint application should be evaluated more quickly; (6) the possibility of reclaiming paint from sludge in water-washed booths should be examined; and (7) more use should be made of film thickness measurements.—R. A. R.

Surface Treatment of Iron and Other Metals by Painting and Similar Preparation. S. Andersen. (*Jernindustri*, 1951, **32**, Apr., 58–60). [In Norwegian]. The author reviews methods of cleaning and preparing metals before painting.

Thoughts on Submarine Paints. H. Rabaté. (*Peintures, Pigments, Vernis*, 1951, **27**, Oct., 619–628). This is a comprehensive discussion of the various aspects of submarine paints, including sections on anti-fouling and anti-corrosion agents.—T. E. D.

Lead Cyanamide. J. H. De Flieger. (*Chem. Week.*, 1951, **47**, Nov. 3, 831–833). [In Dutch]. The properties and advantages of lead cyanamide as an anti-corrosion pigment are surveyed.—R. S.

Lead Cyanamide. N. Kopyloff. (*Peintures, Pigments, Vernis*, 1951, **27**, Nov., 695–696). Some results of the use of lead cyanamide paints are given, and the mechanism of its operation as a corrosion inhibitor is described.—T. E. D.

POWDER METALLURGY

Looking Forward in Powder Metallurgy. H. W. Greenwood. (*Machinery*, 1951, **79**, Aug. 9, 236–237). Various aspects of powder metallurgy are mentioned including the use of coated powders.—E. C. S.

Trends in the Development of Powder Metallurgy. P. Schwarzkopf. (*Schweiz. Arch., angew. Wiss. Tech.*, 1951, **17**, June, 161–170). The development of powder metallurgy during the last 50 years is traced. The latest stage is the production of complex shaped parts by sintering powdered iron or steel. Recent developments in the sintered carbide field have been the production of scale-resistant mixed carbides. Another development still in its early stages is the sintering together of metal powders with oxides for such applications as gas-turbine blades. With the control which can be exercised over grain size and purity, there is no reason why sintered metals should not be made as strong as castings. The porosity of sintered iron or steel can be reduced by impregnating it with about 15% of molten copper after the sintering and maintaining the temperature at about 1150° C. for 1 or 2 hr. This produces a material of 65–70 kg./sq. mm. tensile strength.—M. R. M.

Scientific Research Relating to Powder Metallurgy. W. Rutkowski. (*Hutnik* (Warsaw), 1951, **18**, July–Aug., 293–297). [In Polish]. Some aspects of research in powder metallurgy are discussed.—V. G.

The Development of Powder Metallurgy. W. Trzebiatowski. (*Hutnik* (Warsaw), 1951, **18**, July–Aug., 275–278). [In Polish]. A short survey of the present state and latest developments in powder metallurgy is given. The production of titanium, titanium alloys, molybdenum, ceramic alloys, and abrasive materials is discussed.—V. G.

Powder Metallurgy. E. Bryjak. (*Hutnik* (Warsaw), 1951, **18**, July–Aug., 279–284). [In Polish]. An historical survey of the development of powder metallurgy is given.—V. G.

A Contribution to the Question of Producing Iron Powder by the Reduction of Crystalline Oxides. E. Pelzel. (*Vereinigte Österreichische Eisen- und Stahlwerke Jahrbuch* 1950–51, 125–128). Two methods of producing iron powder from mill-scale are described, one involving reduction of oxide-coal briquettes in a vertical muffle, the other reduction of the oxides by coke-oven gas while travelling through a furnace. The second method is found economically and technically more efficient; impurity contents, particularly sulphur, are also less in the latter method.—P. F.

Sintered Alloys for High-Temperature Service in Gas Turbines. R. W. A. Buswell, W. R. Pitkin, and I. Jenkins. (Symposium on High-Temperature Steels and Alloys for Gas Turbines 1951, *Iron Steel Inst. Special Report No.* 43, 258–268). A cobalt-base alloy of the Vitallium type (Cr 30%, W6%) of low porosity has been developed by powder-metallurgy methods to provide a comparison of the properties to be expected from an alloy prepared by a straightforward sintering process and a similar cast alloy. The work has shown that a product having very low porosity can be obtained in the absence of a liquid phase during sintering provided that powder characteristics are suitably chosen and adequate control is maintained over the sintering conditions. The room-temperature properties of the sintered product compare favourably with those of the cast alloy, but at high temperatures, although the fatigue properties of the sintered material are encouraging, the creep properties at least above 600° are comparatively low. The possibility of improving high-temperature strength by the introduction of refractory oxides such as thoria is discussed. It is considered that the low strength in creep may be associated with the absence of the intergranular carbide networks found in the cast alloy, and possible methods of introducing carbon into the sintered alloy are described. The sintered alloy is very sensitive to heat-treatment. The effect of ageing on mechanical properties is reported and the significance of crystallographic transformation during ageing is discussed.

The Method of Feed-Volume Determination of Metallic Powders. E. Bryjak and Z. Kwasny. (*Hutnik* (Warsaw), 1951, **18**, July–Aug., 290–293). [In Polish]. The powder to be investigated (100 g.) is poured from a funnel-shaped sieve placed in a vibrator, into a measuring cylinder. For determining the volume of a powder with tapping, the receiver is vibrated and not the funnel. The reproducibility of results is claimed to be very good.—V. G.

Particle-Size Analysis of Metal Powders. C. C. Gregg and B. Kopelman. (*Amer. Soc. Met. Preprint* 32, 1951). A rapid routine method has been developed for engineering control in the making of metal powders. The methods were based on the measurement of the rate of fall of the powder in a liquid, or on the gas permeability.—E. T. L.

Density of Powder Metal Parts Controlled Accurately. A. H. Allen. (*Steel*, 1951, **129**, Sept. 17, 84–85). A brief review is given of the advances in powder metallurgy in recent years. Reference is made to newly developed hydraulic powder compacting presses with a fine degree of adjustment in travel of upper and lower rams which permits the desired final density to be obtained throughout the section. New types of powdered metal bearings with lubrication cavities are described.—M. D. J. B.

The Process of Sintering of Metal Powder. W. Dawihl. (*Schweiz. Arch. angew. Wiss. Techn.*, 1951, **17**, Mar., 91–96). Research has shown that during the sintering process the surfaces of adjacent particles form a boundary layer which is stronger than the normal metal. Below a certain minimum temperature, usually about half the absolute temperature at the melting point, sintering will not normally take place. The addition of small quantities of other materials (*e.g.*, 5% cobalt or titanium carbide added to tungsten carbide powder) often greatly assists the sintering either by allowing it to take place at a lower temperature or by the formation of a denser material.—H. R. M.

The Photography of Metallic Powders. S. D. Ford and B. W. Mott. (*Metal Treatment and Drop Forg.*, 1951, **18**, Aug., 360–362). Various experimental methods of lighting for use in the microphotography of metal powders are described. The most suitable involves the use of a viscous liquid such as glycerine for suspending the powder, and a double lighting system.—P. M. C.

Cold War Puts Heat on Metal Powder. J. Kolb. (*Iron Age*, 1951, **167**, May 3, 95–99). The production of sintered iron driving bands for artillery shells is discussed. The advantages suggested include the saving of copper and reduced machining requirements. A tensile strength of 13,000–15,000 lb./sq. in. together with an elongation value of 3% and a density of 5·5 to 5·8 g./c.c. are considered satisfactory.—A. M. F.

The Behaviour of Sintered Iron on Nitriding. W. Köster and J. Ratfelsieper. (*Archiv. Eisenhüttenw.*, 1951, **22**, Sept.–Oct., 337–341). Prismatic sinters were produced from

four different types of iron powder, and nitrided at 500° C. in ammonia from 3·5 to 15 hr. Rapid nitration of the entire volume of the specimens was observed, the speed of nitration depending upon the density of the specimen and the type of powder used in its preparation. An increase in hardness and volume accompanies progressive nitration. Nitrogen absorption of up to 8% is observed. The formation of nitrides was confirmed by X-rays.—P. F.

British Wire Gear Maker Adds Powder Shop. J. Rigby. (*Iron Age*, 1951, **167**, June 21, 90–91). This article gives a brief review of the current drawing, pressing, and sintering practice at J. Rigby and Sons, Ltd., Lowmoor, England. On small complicated parts, powdered metal has cut production costs by 50%.—A. M. F.

PROPERTIES AND TESTS

Testing and Computation of Static Tensile-Shear Load of Spot-Welded Joints. H. Zschokke and R. Montandon. (*Brown Boveri Rev.*, 1950, **37**, Aug.–Sept., 318–334). All calculations of a spot-welded joint subjected to static tensile-shear stress inevitably lead to a problem of structural strength in which changes in the base metal set up by the applied heat must be taken into account. Equations for the breaking load can be derived from the geometry of the specimen and the strength of the sheet. Calculations prove that for every joint there are economical dimensions. These can be applied both when fixing the dimensions of specimens and when designing joints involving rows of spot welds. The method proposed also permits breaking loads to be predicted.

Toughness and the Transition Zone of Steels. L. Hurtado Acera. (*Inst. Hierro Acero*, 1951, **4**, July–Sept., 220–226). [In Spanish]. The concepts of ductility, toughness, tensile strength, and of the temperature of transition from tough to brittle fracture are defined. The relationships between Izod and Mesnager tests are discussed. Finally, some tests are described the object of which was to correlate the values obtained on the large Charpy bar 30 × 30 × 160 mm. with those on the 30 × 10 × 160 mm. bar.—R. S.

The Hot-Torsion Test for Assessing Hot-Working Properties of Steels. D. E. R. Hughes. (*J. Iron Steel Inst.*, 1952, **170**, Mar., 214–220. [This issue].

Investigation of the Resistance to Deformation of Metals and Alloys. G. Wallquist. (*Jernkontorets Ann.*, 1951, **135**, No. 6, 197–218). [In Swedish]. Static and dynamic indentation tests on steels and alloys of aluminium and copper indicate that materials of high hardness, whether due to composition or to low temperature, are more easily deformed by impact than by static pressure. The reverse holds for soft materials. Simple deformability tests are suggested.

A Survey of Some Recent Researches in Theory of Elasticity. J. N. Goodier. (*Appl. Mech. Rev.*, 1951, **4**, June, 330–332). This is a brief survey of recent work on the theory of elasticity. Short sections are devoted to cavities, holes and notches, circular and straight stiffeners, mixed boundary values, contact stress, torsion, flexure, pressure bands on cylinders, and thermal stress. A final section deals with finite strain, initial stress, and stability.—D. H.

Fatigue Fracture of Steel. T. Yokobori. (*J. Phys. Soc. Japan.*, 1951, **6**, Mar.–Apr., 81–86). The causes of the scatter of fatigue-test results using similar specimens under the same conditions are examined and formulæ are developed to express the relationships involved.—R. A. R.

Fatigue of Coiled Springs: Tests on Heavy Silicon-Manganese Steel Tank Components. W. E. Bardgett and F. Gartside. (*Iron Steel*, 1951, **24**, Aug., 375–379; Sept., 411–416; Oct., 454–458). The authors describe a series of investigations into the endurance of silicon–manganese steel coiled springs, using a radial fatigue spring testing rig. The results show that springs coiled at 1000° C. and coiled direct, or coiled at 1000° C. and reheated before oil-quenching from 900° C., have similar endurances and are markedly superior to springs coiled at 800–850° C. and subsequently reheated and oil-quenched from 900° C. Increasing the time of shot peening generally effects a marked improvement in fatigue properties. Raising or lowering the tempering temperature from the normal figures of 470° C. lowers the endurance.—G. F.

The Size Effect in Fatigue of Plain and Notched Steel Specimens Loaded under Reversed Direct Stress. C. E. Phillips and R. B. Heywood. (*Inst. Mech. Eng.*, Feb. 23, 1951, Advance Copy). The faitgue strength under reversed direct stress was ascertained for specimens with diameters from 0·19 to 2·4 in. using steels of 25 and 65 tons/sq. in. tensile strength. No intrinsic size effect was observed with plain specimens. Appreciable size effects were observed with transverse-hole specimens from both steels.—R. A. R.

Automatic Stopping Devices Speed Fatigue Tests. (*Steel*, 1951, **129**, July 16, 78). A description is given of devices recently constructed at the National Bureau of Standards for: (1) Stopping testing machines when a small crack forms in a specimen; and (2) the uniform polishing of fatigue test specimens. Details of a machine for fatigue testing thin sheet specimens in bending are given.—M. D. J. B.

Experimental Study of Plastic Deformation in Metallic Crystals. C. Crussard. (*Métaux-Corrosion-Indust.*, 1951, **26**, July–Aug., 279–291). Recent information, including the author's own contribution, on plastic deformation, is discussed, with particular reference to aluminium and zinc. When the temperature is below a critical value slipping occurs in local planes. Cross-slip was studied optically, and with an electron microscope. The ways in which slip may proceed, with thickening or multiplication of the lines, are described, with regard both to single crystals and to polycrystals. The connection between stress and elastic limit, and between stress and extent of deformation beyond the elastic limit, are dealt with. It is thought that defects in the crystal, or impurities, where traces of alloys are present, constitute the dominating factor. Creep in single crystals is described briefly, and it is shown that for polycrystals the creep is a minimum for a certain grain size.—N. MCE.

Mutual Relationships between the Stress Gradient and the Deformation Gradient. Part II—Effect of Shape and Dimensions. F. Bollenrath and A. Troost. (*Arch. Eisenhüttenw.*, 1951, **22**, Sept.–Oct., 327–335). Part I dealt with plastic distortion (*see J. Iron Steel Inst.*, 1951, **168**, June, 211). In Part II simple relations are derived for the effect of non-uniform stress and strain distributions on plastic flow under diverse types of applied stress, such as bending and torsion of prismatic rods. The characteristic parameters are determined experimentally by means of a tensile test and two other tests, such as bending and torsion, carried out on two geometrically similar specimens.—P. F.

Superposition of the Three Externally Applied Stress Systems Acting on a Member Subject to Bending. O. Föppl. (*Metalloberfläche*, 1951, A, **5**, Jan., A8–A13). A particular solution of the problem of surface stresses in bent beams is advanced. This problem has so far been wrongly understood and no general solution has been obtained.—P. F.

Brittle Fracture of Mild Steel. T. S. Robertson. (Brit. Assoc.: *Engineering*, 1951, **172**, Oct. 5, 445–448). This paper discusses tests at the Naval Construction Research Establishment on brittle fracture in mild steel. Studies of accounts of catastrophic failures in bridges and ships indicate that there are two stages in such failures—First, somehow, the crack must be started; second, this crack must propagate with extreme speed through the structure. This propagation may take place at low nominal stress of about 5 tons/sq. in. Crack propagation and the effects of temperature and fatigue are discussed in detail. Descriptions of the tests and equipment are given.—M. D. J. B.

Cleavage Fractures of Ship Plates. W. M. Wilson, R. A. Hechtman, and W. H. Bruckner. (*Illinois Univ., Eng. Exper. Stat. Bull.*, 1951, Series No. 388, 1–95). An investigation was made to determine the factors influencing the formation of cleavage fractures in ship plates. Fracture characteristics, distribution of strain across plates, relation between load and elongation, factors affecting the strength and energy absorbing capacity of wide plates, transition temperatures, and thickness of plate after fracture are all studied. The tensile strength at room and other temperatures, the chemical compositions of plate steels and hardness surveys of fractured wide plates are discussed. Descriptions of tests are given.—M. D. J. B.

Interpretive Report on Box Girders and Small Specimens. LaMotte Grover, E. M. MacCutcheon, W. S. Pellini, W. Spraragen and J. Vasta. (*Weld. J.*, 1951, **30**, July, 321s–326s). This is an investigation on the effect of a combination of high residual stresses with severe constraint against ductile behaviour, upon the capacity of a structural member to resist rupture under external load. The extent of deformation occurring when a member is subjected to successively increased loads with the load relaxed between each increment was also determined. A highly restrained structure made of fully killed

steel can operate safely under static stresses even at low winter temperatures if it is subjected to only moderate notch effects.

The Measurement of Tension in Problems of Elasticity and Torsion at the Photoelasticity Laboratory of Liège University. A. Pirard. (*Rev. Techn. Luxembourg.*, 1951, **43**, July–Sept., 144–147). Two methods of observing, by polarized light, the tensions and compressions set up in a transparent plastic model under load are described. In the first, white light illuminates the specimen, which is observed through a crossed polarizer and analyser. Many photographs are taken whilst rotating the axis of polarization. These are then projected together. The second method uses a monochromatic circular polarized light, one photograph being taken. The application of the techniques to industrial problems is discussed.—B. G. B.

Brittle Lacquers—The Use of Paint for Shear Testing. R. H. Warring. (*Indust. Finishing*, 1951, **4**, Sept., 133–140). The technique of determining stress distribution with brittle lacquer coatings is explained.—E. T. T.

Use of the Phenomenon of Relaxation of Stresses to Interpret Results of Tests of Railway Car Wheels. O. M. Sidebottom. (*Proc. Soc. Exper. Stress Anal.*, 1951, **9**, 1, 19–26). The author discusses the mechanism of wheel fracture, caused by the heating of the tread by brake-shoe application. Detrimental tensile stresses may be set up by plastic deformation of the tread, followed by cooling. Based on a concept of relaxation of stress at high temperature, a theory is developed to evaluate qualitatively the variables affecting wheel fractures. Experimental work is also described. Residual compressive stresses produced in the rim by heat-treatment, and minimum plate thickness compatible with load carrying requirements, were the factors having the greatest influence on resistance to fracture.—P. M. C.

Contribution to the Calculation of Stresses from Photoelastic Values. D. Vasarhelyi. (*Proc. Soc. Exper. Stress Anal.*, 1951, **9**, 1, 27–34). The author outlines a new method for calculating specific stress values across general sections of photoelastic models. The method has been developed from the integration of the Lamé-Maxwell equation, and involves the summation of alternate elements of principal stress Δp and Δq on trajectories which intersect the section under consideration—P. M. C.

Stresses in Rotating Disks Due to Non-Central Holes. K. E. Barnhart, jun., A. L. Hale, and J. L. Meriam. (*Proc. Soc. Exper. Stress Anal.*, 1951, **9**, 1, 35–52). The stress-freezing photoelastic technique, using the new material Fosterite, has been applied to investigate the effects of non-central holes in balanced rotating discs for a considerable range of sizes, locations, and configurations.—P. M. C.

Microhardness of Constituents in Steel Tested. H. A. Unckel. (*Iron Age*, 1951, **168**, Aug. 30, 68–71). A Hanemann microhardness tester, made by Zeiss has been used to determine the microhardness of ferrite, pearlite, and cementite in several commercial steels. Ferrite in low and medium carbon steels containing 0·25% Mn and 0·50% Si measured 170 diamond hardness number (Dhn). Pearlite in commercial steels of 0·35% C, 0·45% C and 0·60% C measured 310, 320, and 315 Dhn respectively.—A. M. F.

Practical Heat Treatment Stress Relieves Stampings. V. de Pierre and H. Bernstein. (*Steel*, 1951, **129**, Oct. 22, 88–96). The authors describe how the discovery of cracks in several one-piece steel stampings stored for some time led to research on the effects of low temperatures. Severe cold working can invest the mtal with residual stresses of sufficient magnitude to cause splitting over a peiod of time. The beneficial effects of stress-relieving are underlined.—M. D. J. B.

N.P.L. Investigate Rockwell Hardness Test. C. E. Phillips and A. J. Fenner. (*Machinist*, 1951, **95**, Aug. 11, 1183–1189). Variations of up to 2 or 3 units of Rockwell hardness may arise between indenters conforming to the British Standard specification tolerance on tip radius. It is probable that many of the conical diamond indenters produced and employed in industry do not conform to the tolerance limits of radius and angle laid down in B.S. 891 : 1940. The construction of a special testing machine is outlined; the optical comparator and its calibration are described. Radius tolerance, effect of tip profile, range of variation of penetrator profiles, possible effect of creep, estimated changes in hardness reading resulting from variations in tip radius and angle, and effect of changes in load are dealt with.—E. C. S.

The Deformation and Ageing of Mild Steel : II. Characteristics of the Lüders Deformation. E. O. Hall. (*Proc. Phys. Soc., B.*, 1951, **64**, Sept., 742–747). Factors which influence the appearance of Lüders bands in mild steel are studied. It is shown that the Lüders band is adequately described by a uniform shear front, spreading over the specimen. In coarse-grained specimens, experiments indicate that this front becomes diffuse; diffuse bands are also present in strain-aged material, but here the diffuse fronts become sharper as the ageing becomes progressively longer.—C. J. B. F.

The Deformation and Ageing of Mild Steel : III. Discussion of Results. E. O. Hall. (*Proc. Phys. Soc., B.*, 1951, **64**, Sept., 747–753). An attempt is made here to explain the observed phenomena in the yielding and ageing of mild steel, described in Parts I and II, in the general terms of a grain-boundary theory. On this hypothesis, a satisfactory explanation of the variation of the lower yield point with grain size may be developed. It is shown that strain-ageing must involve two processes: (1) A healing of the grain-boundary films, and (2) a hardening in the grains themselves. A discussion of the possible nature of the grain-boundary film is also undertaken.—C. J. B. F.

Strain Aging Effects. J. D. Lubahn. (*Amer. Soc. Met. Preprint* 25, 1951,). This paper includes a review and the results of tensile tests on copper, an aluminium alloy, and steel. The evidence suggests that strengthening of a strained metal during heating, the appearance of a yield point after the heating, discontinuous yielding, and an abnormally low sensitivity to the loading rate all arise together.—E. T. L.

Strain Aging of Pressure Vessel Steels. H. Thielsch. (*Weld. J.*, 1951, **30**, June, 283s–290s). This is a report on work carried out by the Pressure Vessel Research Committee of the Welding Research Council. It was found that strain-ageing embrittlement may be successfully circumvented by using specially killed steels. If susceptible steels are used, the embrittlement effects of strain ageing after cold working may be removed by heat-treatment at or above the recrystallization temperature. Heating at 595° C. for 1 hr. per inch of plate thickness appears to be satisfactory. (45 references).—U. E.

Aluminium-Bearing Non-Ageing Steels. H. Hauttmann. (*Vereinigte Österreichische Eisen- und Stahlwerke Jahrbuch* 1950–51, 37–60). If the aluminium content exceeds 0·02%, and the cooling rate in the A_1 region, after normalizing, exceeds 10° C./min., non-ageing mild steels are obtainable. Numerous experimental test results are given in great detail.

Crack Testing of Important Transmission Parts in Railway Material. A. Janke. (*Jernindustri*, 1951, **32**, July, 102–105). [In Norwegian]. The old method of using a lime wash to detect fatigue cracks in steel has been abandoned at the railway works in Trondheim, and replaced by the Americna Magnaflux and Magnaglo systems, the principles of which are given. A magnetic field is induced in the surface of the test piece and luminescent iron powder dispersed in a liquid is applied, showing the fissures when exposed to ultraviolet light.

Ferromagnetic Properties and Superlattice Formation of Iron–Nickel Alloys. S. Chikazumi. (*J. Phys. Soc., Japan*, 1950, **5**, Sept.–Oct., 327–338). In an earlier paper the author reports investigations on domain-fixing phenomena and their relation to the superlattice formation. In this paper he reports the results of measurements at high temperatures, made to see how the magnetic properties vary during cooling. The mechanism of domain-fixing is also discussed on the basis of the results. A possible explanation of these phenomena may be found by assuming the existence of 'directional order.' A test to prove its existence has been made.—J. P. S.

Effect of a Magnetic Field on the Propagation of Sound Waves in a Ferromagnetic Material. J. De Klerk. (*Nature*, 1951, **168**, Dec. 1, 963–964). For high-frequency longitudinal acoustic waves, a magnetic field a modifies the velocity, due to a change in Young's modulus b and causes a reduction in the attenuation of waves, as the field increases, to a minimum at saturation values. Curves of $\mu_0-\mu_H$ against field strength H are given where μ is an attenuation coefficient.—A. G.

The Change in Thermal E.M.F. Produced by Magnetization of a Single Crystal of Iron. Z. Funatogawa. (*J. Phys. Soc. Japan*, 1950, **5**, Sept.–Oct., 311–316). An experiment is described the purpose of which was to find the dependency of magneto-thermoelectric force on the direction in which a single crystal at an ordinary temperature (25° C.) is magnetized and to give fundamental help in the consideration of the mutual interaction between $3d$ and $4s$ electrons.—J. P. S.

Choosing Equipment for Non-Destructive Testing. C. H. Hastings. (*Amer. Found. Soc. Preprint No.* 15, 1951).

Methods considered are radiography, magnetic particle tests, penetrant tests, and ultrasonic tests. Factors affecting the choice and strength of the radiographic source, such as penetrating power, flaw detectability, and portability or convenience of use are discussed. Magnetic particle tests are considered in a similar way. Both echo and resonance techniques are described with references to their particular advantages and applications.—P. C. P.

Ultrasonics and Their Applications in Technology. H. Thiede. (*Z. Erz. u Met.*, 1951, **4**, June, 212–221). A survey of modern methods of generating ultrasonic vibrations, and of the physical principles underlying them is given. The applications of ultrasonics such as their use in flaw detection, continuous casting, and coagulation of smoke particles are discussed—P. F.

Hydrogen Embrittlement of Steel—Review of the Literature. R. W. Buzzard and H. E. Cleaves. (*U.S. Nat. Bur. Standards*, 1951, Circular 511). This review of the literature up to and including 1949, with 1191 references, shows that no quantitative proof has been established that hydrogen is the sole cause of the many defects attributed to its presence, but it is well established that when procedures for maintaining the hydrogen content at a minimum are utilized, certain defects generally ascribed to hydrogen can be eliminated.—R. A. R.

Diffusion of Hydrogen in Iron and Iron Alloys at Elevated Temperatures. P. L. Chang and W. D. G. Bennett. (*J. Iron Steel Inst.*, 1952, **170**, Mar., 205–213). [This issue].

Calcium Cyanamid—A Source of Nitrogen in Steel. (*Steel*, 1951, **129**; Oct. 8, 138–140). The use of calcium cyanamid in steels is described. Small additions of cyanamid can produce grain refinement and materially increase the strength, hardness, and wear resistance of steels. Its application to increase the nitrogen content of resulphurized and/or rephosphorized low-carbon capped or rimmed steels is also examined. The addition of cyanamid considerably improves machinability. It is generally added to ladle or runner.—M. D. J. B.

Boron Engineering Steels. C. M. Parker. (*Steel*, 1951, **129**, Oct. 15, 76–79). The author discusses the production of new steels, their characteristics and commercial uses. The effects of boron as a truly alloying element are considered. The effects of heat-treatment and the 'fading' in hardenability of boron steels which have been heated for a long time before forging are described. When nitrogen is fixed in steel by such elements as titanium or zirconium, the hardenability conferred by boron is preserved.—M. D. J. B.

A Steel Shortage Overcome. (*Chem. Eng.*, 1951, **58**, Nov., 177). Brief details are given of the ELC (extra low carbon) stainless steels developed by the Armco Steel Corp., Middletown, Ohio. These have very good welding properties, prevent intergranular corrosion, and do not contain scarce stabilizing elements. They are intended to replace niobium-stabilized stainless steel.—R. A. R.

Special Steels for Highly Stressed Welded Structures, Particularly High-Pressure Tubes. H. Hauttmann. (*Vereinigte Österreichische Eisen- und Stahlwerke Jahrbuch* 1950–51, 61–71). The development and tests on some fine-grained aluminium-bearing, low-carbon, low-alloy steels carried out at the V.Ö.E.S.T. steelworks are described. Special aspects relating to the production of these steels are explained. Their performance, particularly in pressure tests of welded-tubing was found very satisfactory.—P. F.

Belgian Researches on Chromium–Molybdenum Steels. P. Cohaur. (*Rev. Univ. Min.*, 1951, **7**, Oct., 336–348). The fundamental metallurgical research which has been done on these steels is reported. The use of transformation curves and the results of laboratory experiments at high temperatures are stated. The type of electrode used and the conditions of welding greatly influence the strength of the weld and its resistance to creep and corrosion. The optimum conditions are stated.—B. G. B.

A Survey of the Development of Creep-Resisting Alloys. N. P. Allen. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 1–10). The development of creep-resisting alloys, both ferritic and austenitic, from 1919 to 1939 is briefly described, and a more detailed account is given of the general trend of the researches undertaken after 1939 in Great Britain, America, and Germany, to provide improved materials for use in gas turbines. The alloys that were relied upon in each country are touched upon, and their properties are described in terms of the stresses giving plastic deformations of the order of 0·1% in 1000 hr.

Gas-Turbine Performance and Materials. J. B. Bucher. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 17–23). The paper gives an indication of some pressing metallurgical problems to which the industrial gas-turbine designer would like answers. The information available today on creep resistance may satisfy the aircraft-engine designers, but it is not sufficient for the design of industrial plants where a life of 100,000 hr. may be required. Long-term tests at comparatively low stresses would be most valuable, particularly for steels suitable for drawing into tubes for the air heaters of closed-cycle gas turbines. More information on weldability and the creep properties, after cold working, of various high-temperature alloys is required before these alloys can be used to their fullest capacity in industrial gas turbines. Corrosion of heat-resisting alloys, due to vanadium pentoxide, has become a serious problem in gas-turbine work. Examples of this are given both for open-cycle plant where the turbine blades were attacked, and for a closed-cycle arrangement where the air-heater tubes were severely corroded. The problem is being attacked along various paths, but no satisfactory solution has yet been obtained. Mention is also made of the desirability of developing a comparatively cheap steel, the properties of which would be in the range between the present-day best ferritics and the cheapest austenitics.

Stresses in Gas-Turbine Discs and Rotors. R. W. Bailey. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 30–35). The author discusses the following points: (1) The nature of the operating stresses in 'thin' and 'thick' discs. (2) Bursting stresses as influenced by the characteristics of ferritic and austenitic steels. (3) The importance of plastic strain in the case of austenitic steel discs, to improve the properties at the central region of a disc or rotor, and thereby to raise its bursting speed. (4) Need to prove the quality of material at the interior of a disc or rotor if the highest working stresses are to be used safely.

Nickel–Chromium–Titanium Alloys of the 'Nimonic 80' Type. L. B. Pfeil, N. P. Allen, and C. G. Conway. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 37–45). A review is given of the information on nickel–chromium-base alloys, which, in 1939, provided a background for development of alloys required to operate at the high temperatures and stresses involved in gas-turbine service. Survey of the literature, supported by unpublished laboratory data and service experience, indicated the potential usefulness of nickel–chromium alloys modified by additions of small amounts of other elements, and subjected to appropriate heat-treatment. Preliminary experiments showed the nickel–chromium–titanium series to be the most promising group. An account is given of research methods used to determine the solid solubility of selected addition agents, the response of the alloys to heat-treatment, their forgeability, and their high-temperature properties. Criteria adopted to assess suitability for high-temperature service are recorded. The work described resulted in the development of 'Nimonic 80,' which was first produced on a commercial scale in 1941. The composition and structure of Nimonic 80 and 80A are discussed in relation to their high-temperature mechanical properties and creep-resistance. Data for the evaluation of properties under varying conditions of temperature, stress, and time are discussed, as affecting the use of the alloys under conditions other than those for which they were originally developed.

Some Proven Gas-Turbine Steels and Related Developments. D. A. Oliver and G. T. Harris. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 46–59). An outline is given of developments by Wm. Jessop and Sons, Ltd., in special steels for jet engines. Their subsequent extensive use in land and marine gas turbines is illustrated. The austenitic steels *R*20 and *G*18*B* are discussed and new long-time creep data presented for times up to 30,000 hr. The ferritic steels are reviewed, including *H*40 and *H*46, and evidence adduced to demonstrate the creep-strength superiority of the stainless *H*46 type up to 630° C. Some considerations governing the

choice and use of ferritic steels are listed. Note is also taken of steels for turbine castings and relaxation-resisting bolts. The influence of gas-atmosphere composition on the oxidation and scaling of different steels is summarized and the comparative immunity of the cobalt-rich *G*32 steel to attack by fuel ash containing vanadium pentoxide is pointed out. The paper concludes with observations on factors governing progress, current trends in development, and a careful summary of the measured low- and high-temperature properties of some widely used steels.

Effect of Warm-Working on an Austenitic Steel (*G*18*B*). G. T. Harris and W. H. Bailey. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 60–67). The problems of warm-working *G*18*B* steel both on an experimental basis and in production have been discussed and the resulting mechanical properties given. Warm-working improves the room-temperature tensile properties and the creep strength, and the higher proof stresses permit the use of higher bore stresses in turbine discs. The effect of turbine disc size on the actual properties obtained is discussed and methods are considered for the partial warm-working of large discs. Future developments of the warm-working process outside the gas-turbine disc field are visualized.

Development of a High-Temperature Alloy for Gas-Turbine Rotor Blades. G. T. Harris and H. C. Child. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 67–80). The general effect of the carbide-forming elements W, Mo, Nb, V, and Ti, the carbon content, and the composition of the base, on the creep strength at 750–800° C. of austenitic Ni–Co–Fe–Cr alloys, has been investigated. The optimum creep strength was found to occur when there were at least three carbide-forming elements present, when the carbon content and the total percentage of carbide-forming elements were carefully balanced, and when the base of the alloy was rich in cobalt. With some practical considerations and these principles in mind, a further investigation was carried out to develop an alloy suitable for gas-turbine rotor blades. The result is Jessop *G*32, a cobalt-base alloy. Some additional work was carried out to determine the optimum heat-treatment for this alloy, which proved to be oil-quenching from the solidus after a 10-min. soak and fully ageing in the temperature range 750–800° C. The nature of the phases which precipitation-harden some of the alloys has been investigated by the carbide-extraction technique.

Properties of Materials Intended for Gas Turbines. H. W. Kirkby and C. Sykes. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 81–94). The paper deals with the properties of creep-resisting steels suitable for use in the gas turbine and jet engine. Materials are classified under (*a*) ferritic steels and (*b*) austenitic steels. The data presented include results of room-temperature mechanical tests and creep tests on blading, bar, and forgings. These data enable comparisons to be made of the intrinsic properties of the various materials and in addition provide information as to the variation in properties with size.

Study of the Properties of a Chromium–Nickel–Niobium Austenitic Steel. H. W. Kirkby and C. Sykes. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 95–106). The paper deals with the properties of wrought 18/10 chromium–nickel steel (stabilized with niobium), with particular reference to high-temperature applications. A large part is devoted to the properties of bar, but a section is included on forgings, and comparisons are made. Data presented comprise creep properties representing testing times up to 20,000 hr. at 550–700° C. inclusive, together with short-time tensile test results. In addition, data are given on room-temperature tensile properties, as influenced by exposure at temperatures of 600° and 650° C. Fatigue properties are also discussed. The various metallographical aspects of the 18/10 type of austenitic steel, in regard to stability and other structural changes involved after exposure at temperature (600–850° C.), have been investigated. The data given include the findings on sigma-forming tendencies of the steel and the metallographic techniques used. Some attention has been given to the influence of heat-treatment on creep resistance at 650° and 700° C., coupled with the influence of grain size *per se* on creep properties for a temperature of 650° C. The factors which may influence the shape of the creep curves obtained are discussed.

Creep-Resisting Ferritic Steels. E. W. Colbeck and J. R. Rait. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 107–124). A brief historical review of the demand for creep-resisting ferritic alloy steels including details of such steels employed in the early development of gas turbines is given. One of the outstanding materials of this type, namely, the 3% Cr–Mo–W–V alloy steel has been studied in great detail in an attempt to evaluate the influence of such important factors as composition, constitution, and heat-treatment on the creep properties. The hardened and tempered steel consists of an aggregate of ferrite and carbides, the composition of the former and the type and amount of the latter exerting important influences on the creep properties. An extension of the investigation to include a series of alloys based on the 3% Cr–Mo–W–V type in which the chromium is varied from 0 to 12% provides further valuable information about the influence of composition, constitution of the ferrite–carbide aggregate, and heat-treatment on the creep properties. The detailed properties of the 3% Cr–Mo–W–V steel (H.G.T.3) in the wrought condition are provided including a few long-time creep tests. A preliminary investigation of the creep properties of these steels in the cast condition is also included. As a result of this systematic investigation it is believed that at least three other steels in addition to H.G.T.3, with 0, 1·0, and 10·0% chromium contents respectively, with excellent creep properties will become available in the near future.

Ferritic Steels for Gas Turbines. H. H. Burton, J. E. Russell, and D. V. Walker. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 125–134). Test results are presented on three ferrite creep-resisting steels, mainly of the 3% Cr–Mo–W–V type, the test specimens being taken from forged discs as well as from bars. The effect of variations in treatment and mechanical properties on the creep properties is investigated, and data on the effective modulus of elasticity at test temperatures are given. The results are compared in the light of requirements for materials for turbine wheels in aeroplane gas turbines.

Special Steels for Gas Turbines. W. E. Bardgett and G. R. Bolsover. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 135–148). The paper deals with a few selected aspects of the use of special steels for gas-turbine work. Data are given of a steel containing nickel, chromium, molybdenum, cobalt, niobium, wolfram, and titanium, tested both for creep and for general mechanical properties, with different amounts of hot work, and tested transversely and longitudinally. The results show little effect of either direction or amount of work on the essential properties. Properties are presented of a 25% Cr, 15% Ni type steel, suitable for combustion chambers. A 20% Cr, 30% Ni, 1% Ti type steel also suitable for combustion chambers and showing remarkably good creep resistance at certain temperatures, for a steel of such relatively low alloy content, is dealt with finally in the form of a disc. Data on tests on this steel obtained to date on 'negative creep' are included.

Fatigue at High Temperatures. H. J. Tapsell. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 169–174). The need for fatigue investigations of metals at high temperatures is emphasized. Consideration is first given to the general characteristics of the behaviour of metals in fatigue, together with the modifications introduced by the presence of mean steady stresses which cause creep. The effect of plastic strain in modifying the stress distribution under bending conditions is described, and it is shown that cyclic speed of stressing is an important factor to be considered. Experimental fatigue and creep data are given for some turbine-blade materials, and the method of correlating these data in working stress diagrams is shown. The paper ends with short statements regarding investigations into the effect of exhaust gases, small fillet radii, and welds.

Fatigue Tests at Elevated Temperatures. P. H. Frith. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43,

175–181). Results are given of tests to determine the fatigue properties of various heat-resisting materials at elevated temperatures. The tests were carried out on hollow test pieces with reversed bending stresses, and with reversed bending stresses superimposed on a static tension or static bending stress. A few tests were carried out on turbine blades with reversed bending stresses.

Hot Fatigue Testing. H. E. Gresham and B. Hall. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 181–185). This paper describes apparatus designed and constructed by the authors for investigation of fatigue strength of materials at elevated temperatures. General details of the construction of a simple single-point loading fatigue machine are given, and the construction of the furnace and temperature measurement is discussed. The adaptability of the apparatus is illustrated by its use for corrosion-fatigue testing with corrosion media in the form of gases, solids, or as a spray. Specific research items in which this apparatus has proved useful are discussed in some detail, and illustrations are given of a few of the more interesting results.

Variation of Elastic Moduli with Temperature for Various Steels and Pure Metals. G. T. Harris and M. T. Watkins. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 185–188). Determination of Young's Modulus of several gas-turbine materials and a few pure metals at temperatures up to 800° C. have been made by static and dynamic methods. In general the values determined by the two methods agree within 1%. Determinations have been made on two steels (*G*18*B* and *R*20) of the variation with temperature of the modulus of rigidity and Poisson's Ratio.

Chromium-Base Alloys for Gas-Turbine Applications. E. A. G. Liddiard and A. H. Sully. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 243–245). The methods of producing chromium-base alloys are briefly reviewed. Difficulties are encountered in all methods designed to remove oxygen from the metal and to prevent further contamination by oxygen, nitrogen, or carbon. Melting and casting operations must be carried out in a vacuum or in an atmosphere free from these elements. Certain chromium-base alloys possess a very high level of creep properties at 900–1000° C., and are probably superior to any other metallic alloys so far investigated in this range of temperature. They also have the advantage of a high resistance to oxidation and a fairly low density. Unfortunately, chromium and its alloys have extremely low ductility at room temperature; the reason for this has not yet been completely elucidated. The applicability of chromium alloys to gas turbines would be much widened if the ductility at normal temperatures could be significantly improved.

Some Cobalt-Rich Alloys for High-Temperature Service. J. C. Chaston and F. C. Child. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 246–248). The stress-to-rupture characteristics at 900° C. of some alloys in the tantalum–chromium–cobalt system have been examined, particular attention being given to the influence of carbon on the creep endurance and microstructure of alloys containing 10% of tantalum and 10% of chromium. Optimum results appear to be obtained by the addition of about 0·3% of carbon to these alloys, the cast alloy then having a life-to-rupture of about 1500 hr. under a stress of 4·5 tons/sq. in. and 800 hr. under 5·0 tons/sq. in.

Research and Development Work on High-Temperature Materials. C. A. Bristow and H. Sutton. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 289–292). Attention is drawn to the importance of availability in deciding on the constituent elements for high-temperature alloys. The ever-increasing amount of metal in use per head of a rapidly increasing world population calls for the avoidance of misuse, wastage, and loss, especially of less abundant metals like cobalt and niobium. Metallurgical factors affecting the choice of metallic materials are discussed and illustrated by various types of alloy. Long-range fundamental research is essential.

Future Needs in Materials for Land and Marine Gas Turbines. J. M. Robertson. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 293–303). A general idea is given of the large range covered by the many types of land and marine gas turbines that are now being operated, constructed, designed, or considered. Attention is given to the factors that are important in relation to the performance of materials in gas-turbine components that operate at high temperatures, to the making, shaping, and treating of these components, and to their cost. A list is given of the classes of material now being used, and likely to be used in the near future, in the production of gas-turbine components. The conditions under which different components work are described, and the relations between materials and the production and properties of components are discussed.

Design of Plant and Choice of Materials for Refineries. W. P. Kerkhof. (*Rev. Univ. Min.*, 1951, **7**, Oct., 327–336). The author describes the special metallurgical problems which arise in oil-refinery construction. Vessels for use at high (550° C.) and low (−100° C.) temperatures and high (300 kg./sq. cm.) and low (vacuum) pressures have to be built. The selection of a suitable iron alloy is complicated by the corrosion conditions which often arise. Welded construction is widely used; great care must be taken to prevent weld decay. For containers under pressure at between − 35° C. and 400–450° C. high-quality open-hearth steel can be used. At temperatures $<-15°$ C. and up to 350° C., killed steel must be used. For very low temperatures the carbon and hydrogen content should be low. Between 400° C. and 550° C. 0·5% Mo steel is suitable; this steel also must be killed. High chromium (12%) and 18/8 steels are suitable for corrosive conditions. The use of 'clad' steel is described.

Stainless Steels and Other Ferrous Alloys as Chemical Engineering Materials of Construction. W. A. Luce. (*Indust. and Eng. Chem.*, 1951, **43**, Oct., 2258–2271). Information published during the past year on this subject is surveyed. The topics reviewed are: Titanium stabilization and substitution of titanium for niobium; the nature, occurrence, and effects of the sigma phase in stainless steels; corrosion; mechanical properties and structure; high temperature data; welding; manufacture, machining, surface treatment, and applications of stainless steels; stainless-steel powders; high-silicon irons, iron–nickel alloys, and austenitic manganese steels. (328 references).—C. J. B. F.

Iron, Mild Steels, and Low-Alloy Steels as Chemical Engineering Materials of Construction. C. P. Larrabee and B. J. Kelly. (*Indust. Eng. Chem.*, 1951, **43**, Oct., 2239–2242). Information published on this subject during the past year is summarized. Topics reviewed are: Irons and iron powder, particular attention being drawn to the increasing use of spheroidal cast iron; mild steels, their corrosion, and the cathodic protection of iron and steel installations; alloy steels; and high-strength low-alloy steels. (49 references).

The Physical Properties of the Nickel-Iron Alloys. (*Mond Nickel Co. Ltd. Pamphlet*, 1951, 1–22). The history of investigations made on the nickel–iron system is surveyed, and the general characteristics of the system are given. The dilatation properties of the binary alloys are dealt with in detail, with particular reference to the 36% Ni–Fe alloy and its applications where a material with a low coefficient of expansion is required. The ternary alloys Ni–Fe–Co and Ni–Fe–Cr, are also mentioned; their most important application is that of glass-to-metal seals. Finally the 'constant-modulus' alloys are reviewed.—T. E. D.

Instruments Unsnarl Metal Mixups. A. Doschek. (*Iron Age*, 1951, **167**, June 21, 92–96). Magnetic methods for sorting are described and in this field permeability has the biggest effect, but the method is dependent on uniform size of test pieces. A new instrument operating on the triboelectric effect, that is the current generated when two alloys of dissimilar metallurgy are placed in frictional contact. Advantages of this method lie in its ability to sort both ferromagnetic and non-ferromagnetic alloys and test pieces of any size.—A. M. F.

Low Temperature Steel Processing. T. A. Dickinson. (*Steel Processing*, 1951, **37**, Aug., 381, 399). The author suggests that sub-zero temperatures may eventually find more and more uses in steel and metal treating processes. The phenomena of super-conductivity, loss of magnetic properties, and efficient rectification of alternating current, exhibited by various metals at very low temperatures, are mentioned.

Recent Developments in Mechanics and Strength of Materials. F. K. G. Odqvist. (*IVA*, 1946, **17**, 5, 200–202). [In Swedish].

Paul's theory of the relationship between magnetic permeability and mechanical properties of steels is criticized. Papers presented at the 6th International Congress on Applied Mechanics are examined.—B. S. E.

The Magnetic Properties of the Nickel–Iron Alloys. (Mond Nickel Co., Ltd., 1949). The development of Ni–Fe alloys is reviewed. Alloys containing about 30% Ni are normally non-magnetic and possess Curie points near to or below room temperature. Alloys of the Permalloy type (35–80% Ni), exhibit high permeabilities at low flux densities, low hysteresis loss, and high corrosion resistance, and are easily worked. The permanent magnet alloys (<30% Ni) with aluminium as an essential addition, exhibit high remanences and fairly low coercive forces. (55 references).—J. G. W.

The Influence of Compression and Fritting on the Magnetic Susceptibility of Iron-Nickel Powders. L. Weil. (*J. Phys. Radium*, 1951, **12**, Apr., 520–526). This paper describes experiments between −193 and +300° C. showing that the magnetic susceptibility of powders that have not been treated, and where anisotropy controls domain alignment, usually obeys Neel's laws. Explanations in terms of the formation of Bloch walls and Brownian movement, are advanced to account for the difference with heated agglomerates.—E. T. L.

The Thermal Conductivity of Some Alloys at Low Temperatures. R. Berman. (*Phil. Mag.*, 1951, **42**, June, 642–650). The thermal conductivities of German silver, stainless steel, and constantan have been determined between 2° and 90° K. and the electronic and lattice components of the conductivity have been calculated. A table gives the heat flow along specimens of each alloy.—J. P. S.

Resistivity of Ferrites of Zinc, Nickel, Cobalt, Magnesium, and Copper as a Function of Temperature. L. Bochirol. (*Compt. Rend.*, 1951, **233**, Oct. 1, 736–738). Resistivities are studied in the temperature range 100°–700° C. and the effect of thermal treatment noted. Quenching from 700° C. or above causes a considerable lowering of the resistivity.—A. G.

Application of Artificial Radioactive Sources to Radiographic Metal Testing. C. Brachet. (*Métaux-Corrosion-Indust.*, 1951, **26**, May, 205–213). Artificial radioactive sources, besides being cheaper than natural sources, have the advantage of greater convenience than X-rays, where great accuracy or speed is not needed. The most useful isotopes are those of cobalt, tantalum, and iridium; their relative merits for different applications, and the necessary safety precautions, are discussed. Salient points in connection with their use, such as the penumbra due to the size of the source, the effect of distance, and the type of film are described.—N. MCE.

New Developments in Xeroradiography. R. C. McMaster. (*Non-Destructive Test.*, 1951, **10**, Summer Issue, 8–25). Xeroradiography is a recently developed dry radiographic process, in which a metal plate carrying a thin photo-sensitive film is sensitized by the deposition of a uniform static electric charge. On exposure to X-rays, the charge leaks away to the metal backing plate and a latent electrostatic image is produced. This image can be made visible by various pigmented powders, and permanent records may be obtained by transferring the powder image to paper. The plate may be cleaned, recharged, and used again indefinitely. The complete process is reviewed, the theory of semi-conductors is outlined, and the response of the process to X-rays in the range 5 to 10,000 kV. (peak) is examined in great detail.—P. M. C.

Intensification of Underexposed Industrial Radiographs. G. M. Corney and H. R. Splettstosser. (*Non-Destructive Test.*, 1951, **10**, Summer Issue, 29–32). In many cases, underexposed industrial radiographs can be salvaged by treatment with Kodak Intensifier In.-6. The intensification process is fully described, the solutions are given, and the speeds, contrasts, and fogs of several types of film after treatment are outlined. Results of actual intensification of purposely underexposed films are shown.—P. M. C.

Gamma-Rays for Non-Destructive Testing. C. G. Carlström. (*Gjuteriet*, 1951, **41**, May, 67–71). [In Swedish]. The versatility and comparatively low costs of gamma-radiography in conjunction with the small size of the radiation emitter make the method widely applicable. Examples using Co^{60} and Ir^{192} are quoted.—B. S. E.

Non-Destructive Testing of Large Forgings by Supersonic Vibrations. W. Felix. (*Schweizer Archiv Wiss. Techn.*, 1951, **17**, Apr., 107–113). Supersonic testing is particularly useful for large forgings of simple form. In general, it cannot be used if the thickness is less than 15 mm. Examples of its use are described, the equipment employed for the tests being a Hughes Supersonic Flaw Detector which transmits a short impulse of vibration (2–5 microsec.) every 0·02 sec. and records both the impulse and its reflections on a cathode-ray tube.—H. R. M.

Non-Destructive Testing of Materials by Radioactive Isotopes. The Gamma-ray Apparatus. E. Mittli. (*Betrieb u. Fertigung*, 1951, **5**, Mar., 45–50). Certain radioactive isotopes now available (*e.g.*, iridium 192, tantalum 182, cobalt 60) can be used as a source of gamma-rays for non-destructive testing. They occupy much smaller volume than the equivalent quantity of radium bromide and thus give radiography with better definition, while they have the advantage over radon of lower cost and much longer life. They are particularly useful for testing thick sections (*e.g.*, over 60 mm. of steel) or components having sections of widely differing thickness, which are outside the range suitable for X-ray testing. The contrast is less, but the ability to distinguish details better, than with X-rays. A portable heavy-metal container for isotopes, about 23 × 25 × 28 cm. weighing 17 kg., has been designed.—H. R. M.

Methods of Measuring the Thermal Conductivities of Solids. P. Vernotte. (*Métaux-Corrosion-Indust.*, 1951, **26**, May, 216–217). For good conductors, a method is outlined in which the specimen is a long bar along which a sinusoidal thermal wave is transmitted, the resulting wave being subjected to a Fourier analysis. In the author's modification, the wave need not have an imposed form. For poor conductors, the heat transferred to a metal block touching the specimen for a known time can be measured.—N. MCE.

Low Temperature Properties of Ferrous Materials. (*Soc. Automotive Eng. Spec. Publ.* 65, 1950). A detailed survey is given of published literature, mainly American, relating to the properties of plain carbon and alloy steels at sub-zero temperatures. (42 references).—J. G. W.

On Various Methods of Testing Alloys Mechanically Resistant to Heat. A. Gueussier. (*Métaux-Corrosion-Indust.*, 1951, **26**, July–Aug., 310–317). The classical methods of studying the plastic properties of metals at high temperatures are discussed. Creep is difficult to measure accurately, and time-deformation tests may be very protracted. To illustrate how measurements of hardness may be used, chromium–nickel steels are taken as examples, and the inter-relationships of load, temperature, tungsten additions, and recrystallization temperature are shown.—N. MCE.

Study of the Manufacture of Valves for Aviation, Automobile and Diesel Engines—Steels, Special Metals, and Methods of Improving the High Temperature Properties and Avoiding Wear and Corrosion. A. Balleret. (*Métaux-Corrosion-Indust.*, 1951, **26**, June, 256–268). The exacting requirements of exhaust valves demand great care in the manufacture of valves for internal combustion engines. The special steels required are either austenitic nickel–chromium, martensitic silicon–chromium or a recently improved alloy containing molybdenum. To achieve rapid cooling of the stem, the technique of forging them hollow, and heat-treating them by distilling sodium metal from the cavity, is described; they may be finished by polishing or nitriding. It is also explained how the rims may be hardened by inserting a ring of special alloy, with due precautions against grain growth or diffusion: and the heads can be protected from attack by lead oxide by coating with an inoxidizable alloy, such as the British products "Brightray" or "Stellite," and the French "Alacrite 60."—N. MCE.

Room Temperature Creep Tests on Steel Wires. A. Krisch. (*Arch. Eisenhüttenw.*, 1951, **22**, Sept.–Oct., 313–316). Stress-relaxation creep tests, simulating conditions prevailing in pre-stressed concrete, were carried out on patented and oil-tempered steels of 98–146 tons/sq. in. ultimate tensile strength. Stresses ranged from 45 to 97% of ultimate tensile strength. Log. stress–time plots were linear and gave permissible stress relaxations if extrapolated to 100 years.

Effect of Wire Diameter on the Creep of Steel Wires at Room Temperature. E. Uhlir. (*Betrieb u. Fertigung*, 1951, **5**, Aug., 133–136). Constant-stress creep tests were carried out on 0·78% C patented steel wires, 1·48, 1·98, and 3·23 mm. in dia. and length 3 m., under stresses from 0·5 to 0·63 of the tensile strength. The diameters before drawing were 5, 6·5, and 7 mm. respectively. The amount of creep in 150 hr. at 20° C. increased with the wire diameter. At high temperatures, on account of recrystallization, the thin wires showed most deformation.—H. R. M.

Files and File Steels. O. Pattermann. (*Österr. Masch. Elektrowirtschaft*, 1950, **5**, Dec. 15, 484–494; 1951, **6**, Mar. 1, 63–66; Mar. 31, 97–99). Part I describes the different types of file and rasp made and the various ways in which they are classified. Part II describes the production processes, including forging, grinding, cutting of teeth, and heat-treatment. Part III deals with file steels and gives the compositions of carbon and alloy steels and the types of file or rasp for which each is used.—H. R. M.

Special Steels in the Plastics Industry. O. Mirt. (*Betrieb u. Fertigung*, 1951, **5**, July, 109–117; Sept., 145–151). The composition, heat-treatment, and properties of corrosion-resistant steels suitable for constructing plant used in the manufacture of plastics are tabulated. Machines used for the shaping and working of plastics in the fluid, plastic or solid state are described, the required properties of materials for constructing the various working tools are discussed, and suitable ranges of steels having these properties are listed.—H. R. M.

Two New Gear Materials. C. M. Schwitter. (*Machinery*, 1951, **78**, June 21, 1032–1038). The properties and application in gear manufacture of the recently developed ductile cast iron with nodular graphite are discussed.—E. C. S.

The Influence of Production Factors on the Properties of Hot Rolled Transformer Sheets. M. Markuszewicz and J. Kozielski. (*Prace Glow. Inst. Met.*, 1951, **3**, 4, 279–295). [In Polish]. The properties of transformer steels are discussed and opinions on the influence of chemical composition, structure, and stress on the magnetic properties are reviewed. The effects of oxidation with oxygen and ore, and deoxidation under dark and light slags were tested in 35 heats. The relationship between the oxygen in the steel and power losses, and the optimum conditions of refining were determined. The degree of decarburization and the structural changes during rolling were investigated. No appreciable variations in magnetic properties of transformer sheets were found with increase in heating time or lower temperature at the end of rolling. The optimum temperature range of annealing was 780–820° C.; under or overheating caused deterioration of magnetic properties. After annealing in hydrogen at 1100–1250° C. about 20% decrease in power losses was found; this treatment is recommended.—V. G.

New Standards for Tubes for Making Locomotive Piston Rings. P. Januszewicz. (*Przeglad Odlewnictwa*, 1951, **1**, July–Aug., 184–191). [In Polish]. A method for determining the quality of piston rings, and present and proposed technical standards are discussed.—V. G.

Anchoring and Cold Stretching of Reinforcement Bars. C. Forssell. (*Schweiz. Arch., angew. Wiss. Techn.*, 1950, **16**, July, 200–213). The advantages of cold-stretched steel for concrete reinforcement are discussed. Cold-stretching 5–10% leads, after an ageing period, to an increase of 90–100% in the lower yield stress, at a cost of about 3% of the price of the steel. Compared with cold-stretched 0·60% C steel, the ordinary mild-steel reinforcement uses 100–150% more steel and costs 20–50% more. Compared with cold-stretched mild steel (St. 44) these figures become 30–70% and 15–30% respectively. Different methods of anchoring are compared by tests on concrete beams.—H. R. M.

Interrelation of Mechanical Properties, Casting Size, and Microstructure of Ductile Cast Iron. R. W. Kraft and R. A. Flinn. (*Amer. Soc. Met. Preprint* 33, 1951). The authors investigated the variation of mechanical properties in castings of different thicknesses, and correlation of properties of steels with the same matrices, and the reproducibility of properties commercially.—E. T. L.

Nodular Graphite Cast Iron. C. Galletto. (*Riv. Mec.*, 1951, **2**, Sept. 1, 7–14). [In Italian]. The mechanical and metallurgical properties of nodular graphite iron are examined. Whilst the properties and applications of this iron are attractive, its production is not simple. Reliable grades can only be obtained by the strictest quality control and only in well-equipped foundries possessing laboratory facilities.

M. D. J. B.

Ductile Iron Replaces Alloy Gear Castings, Forgings. J. D. Sheley. (*Iron Age*, 1951, **167**, May 10, 99–100). Ductile iron is being used to replace alloy steel castings and forgings. It can be successfully welded to steel, stainless steel, and to itself. Careful heat-treatment can give a range of tensile strength from 65,000 to over 100,000 lb./sq. in. and hardness from 170 to over 450 Brinell.—A. M. F.

The Use of High-Quality Cast Iron as a Constructional Material. J. Weber. (*Przeglad Odlewnictwa*, 1951, **1**, Sept., 241–242). [In Polish]. The author directs attention to the necessity for more research into the properties of high-quality cast iron and for publishing standards to enable engineers to use this material more widely.—V. G.

Sigma Phase in Several Cast Austenitic Steels. V. T. Malcolm and S. Low. (*Symposium on the Nature, Occurrence, and Effects of Sigma Phase: Amer. Soc. Test. Mat.*, 1950, *Spec. Tech. Pub.*, 110, 38–44). Three cast 18/8 and three cast 16/35 Ni–Cr alloys, commonly used for cast valves and fittings, were investigated for the embrittling effect of the sigma phase, after exposure to temperatures of 1000° and 1500° F. This effect was determined by the keyhole-notch Charpy impact test at room temperature and at 1200–1500° F. Sigma-phase formation was only conclusively identified in the case of the 18/8 alloys.—J. B. B.

The Formation of Sigma Phase in 17 per cent. Chromium Steel. J. J. Heger. (*Symposium on the Nature, Occurrence, and Effects of Sigma Phase: Amer. Soc. Test. Mat.*, 1950, *Spec. Tech. Pub.* 110, 75–78). By using cold work and extended heating periods at 566° C., sigma phase has been formed and identified in 17% Cr stainless steel.—J. B. B.

Sigma Phase and Other Effects of Prolonged Heating at Elevated Temperatures on 25 per cent Chromium–20 per cent Nickel Steel. G. N. Emmanuel. (*Symposium on the Nature, Occurrence, and Effects of Sigma Phase: Amer. Soc. Test. Mat.*, 1950, *Spec. Tech. Pub.* 110, 82–99). Studies at elevated temperatures on 25% Cr–20% Ni (type 310) stainless steel have shown that the sigma phase is formed directly from an austenitic matrix, in the temperature range 1100–1600° F., and that previous cold working increases its rate of formation. The rate of formation increased with temperature, reaching a maximum at 1500° F. and then decreased to zero at 1700° F. An initially coarse-grained alloy exhibits resistance to sigma formation. Toughness of brittle specimens was improved during elevated-temperature impact testing. Toughness could be restored to embrittled specimens by heating to 1875° F. and then quenching. Appreciable grain growth in this alloy only takes place above 1800° F.—J. B. B.

The Occurrence of the Sigma Phase and Its Effect on Certain Properties of Cast Fe–Ni–Cr Alloys. J. H. Jackson. (*Symposium on the Nature, Occurrence, and Effects of Sigma Phase: Amer. Soc. Test. Mat.*, 1950, *Spec. Tech. Pub.* 110, 100–119). The sigma phase in the widely used HH alloy (cast 26% Cr–12% Ni type, has been shown to have detrimental effects. A 'thermal fatigue' test, developed to determine hot cracking and distortion tendencies in heat-resisting steels, has shown that sigma-free alloys are much less susceptible to cracking and distortion than sigma-bearing alloys. HK alloy (cast 26% Cr–20% Ni type) is also widely used, and sigma formation can be reduced, as with HH alloy, by composition control. Of the other alloys currently being produced in the form of heat-resistant castings, most contain at least 33% Ni and $\geq$21% Cr, and are thought not to be susceptible to sigma formation.—J. B. B.

Some Notes on the Structure and Impact Resistance of Columbium-Bearing 18-8 Steels after Exposure to Elevated Temperatures. W. O. Binder. (*Symposium on the Nature, Occurrence, and Effects of Sigma Phase: Amer. Soc. Test. Mat.*, 1950, *Spec. Tech. Pub.* 110, 146–164). The influence of columbium on the impact properties has been studied in steels containing 8–16% Ni and 0–3% Cb. The addition of >2% Cb decreases the toughness of the steel in the annealed condition. Long heating at 650° and 870° C. has no effect on impact toughness of steels with up to 2% Cb, but is detrimental to higher columbium steels. X-ray identification of sigma phase was obtained after long periods at 650° C. over the range 8–16% Ni and 0·75–3% Cb. Cold working, followed by low-temperature annealing, accelerates the rate of precipitation of sigma phase and favours particle growth, whereas high-temperature annealing inhibits precipitation and grain growth.—J. B. B.

Observations on the Effect of Sigma on the Mechanical Properties of Columbium-Stabilized Weldments in Austenitic Stainless Steels. F. W. Schmitz and M. A. Scheil. (*Symposium on the Nature, Occurrence, and Effects of Sigma Phase: Amer. Soc. Test. Mat.*, 1950, *Spec. Tech. Pub.* 110, 165–177). A study of 13 weldments in 1½-in. thick T347 stainless steel reveals that the ductility of all weld metal test bars is affected by the following variations: Maximum ductility is obtained

in the as-welded condition with 3–8% ferrite and no sigma present; heat-treatments producing sigma lower ductility; weld metal with ferrite in the as-deposited condition does not always form sigma to the full extent possible; the size and distribution of the sigma phase alters the ductility—J. B. B.

METALLOGRAPHY

Identification and Mode of Formation and Re-Solution of Sigma Phase in Austenitic Chromium–Nickel Steels. E. J. Dulis and G. V. Smith. (*Symposium on the Nature, Occurrence, and Effects of Sigma Phase: Amer. Soc. Test Mat.*, 1950, *Spec. Tech. Pub.* 110, 3–29). Investigation of the etching characteristics of phases occurring in austenitic stainless steels, supplemented by X-ray diffraction and magnetic permeability studies, has suggested procedures by which the sigma, carbide, and ferrite phases can be distinguished, with certain exceptions, under the microscope. The mode of formation and re-solution of sigma was observed, and transient formation of ferrite was detected during the solution of sigma at a temperature above its stable range.

X-Ray Study of the Sigma Phase in Various Alloy Systems. P. Duwez and S. R. Baen. (*Symposium on the Nature, Occurrence, and Effects of Sigma Phase: Amer. Soc. Test. Mat.*, 1950, *Spec. Tech. Pub.* 110, 48–54). X-ray diffraction data of the sigma phase in the binary alloys of iron, cobalt, nickel, chromium, and vanadium are critically reviewed. A tentative tetragonal unit cell is proposed for the sigma phase. On this basis the lattice parameters of the various binary sigma phases are computed. The ternary sigma solid solution in the Fe–Cr–Mo system and the influence of molybdenum on the lattic parameter of sigma are discussed. The relation of atomic diameter and crystal structure of the component metals in binary alloys to the sigma phase is described. An orthorhombic cell may describe the sigma phase.—J. B. B.

Sigma Phase in Chromium–Molybdenum Alloys with Iron or Nickel. J. W. Putman, N. J. Grant, and D. S. Bloom. (*Symposium on the Nature, Occurrence, and Effects of Sigma Phase: Amer. Soc. Test. Mat.*, 1950, *Spec. Tech. Pub.* 110, 61–68). The extent of the sigma phase in the Cr–Mo–Fe ternary diagram has been determined, and some properties of the sigma occurring in this system have been described. Molybdenum increases the high-temperature stability of sigma in Fe–Cr alloys, and the sigma phase in the 60Cr–25Mo–15Fe alloy is stable at 1100° C. In this alloy the tendency for the formation of sigma is increased at low temperatures, provided that sufficient time is provided for the transformation. The sigma phase occurs in the Cr–Mo–Ni ternary alloys, but has not been reported in any of the three constituent binaries. X-ray diffraction data on Cr–Mo–Ni sigma are included.—J. B. B.

The Tetragonality of the Sigma Phase in the Iron–Chromium System. L. Menezes, J. K. Roros, and T. A. Read. (*Symposium on the Nature, Occurrence, and Effects of Sigma Phase: Amer. Soc. Test. Mat.*, 1950, *Spec. Tech. Pub.* 110, 71–74). Single crystal specimens of the 50% Fe–50% Cr sigma phase were prepared. Back-reflection Laue diffraction patterns revealed a four-fold symmetry axis and two twofold axes orthogonal to it and to each other, characteristic of tetragonal crystals with the Laue symmetry 4/mmm.—J. B. B.

The Phase Diagrams of the Ternary Systems Fe–Cr–W and Fe–Cr–Mo at Low Temperatures. H. J. Goldschmidt. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 249–257). The phase-relationships of the iron–chromium–molybdenum and iron–chromium–tungsten systems have been investigated by X-ray analysis for a 600° C. equilibrium, using powder-diffusion methods. The results are discussed, particularly the behaviour of the sigma and Xi phases, which are largely competitive and tend to inhibit each other, and of the primary alpha solid solutions; the latter form miscibility bays with potential precipitation-hardening effects. A ternary compound ('N') occurs in the Fe–Cr–Mo system, of approximate composition Fe_4CrMo_2. The compound FeMo existing at high temperatures (above 1180° C.) in the iron–molybdenum binary, but retainable by quenching, is found to possess the sigma structure; it forms a continuous series of solid solution with FeCr; in contrast to the latter, it is stable up to its melting point. Practical applications of the two alloy systems are pointed out.

Isothermal Transformation of Austenite. A. Hultgren and Co-Workers. (*Jernkontorets Annaler*, 1951, **135**, No. 8, 403–494). [In Swedish]. The distribution in several ternary alloy steels of copper, cobalt, nickel, tungsten, molybdenum, manganese, and chromium between ferrite and cementite at equilibrium has been studied and distribution ratios are reported. An account is given of different reactions occurring during isothermal transformation of alloy steels, and the microstructures have been examined.—B. S. E.

On the Superlattice Formation in Binary Alloys of Body-Centred Cubic Type. S. Matsuda. (*J. Phys. Soc. Japan*, 1951, **6**, May–June, 131–135). In previous theories the formation of superlattice is discussed by taking into account the interaction between atoms on the nearest neighbouring lattice points and the interactions between the next nearest points were considered to have little effect. The present author believes the effect of the latter is important in some cases of body-centred cubic binary alloys; he discusses in particular superlattice formation in the iron–aluminium system.

Structure of Carbides in Alloy Steels. Part II—Carbide Formation in High-Speed Steels. H. J. Goldschmidt. (*J. Iron Steel Inst.* 1952, **170**, Mar., 189–204). [This issue].

CORROSION

Iron/Sulphide Ratios in Corrosion by Sulphate Reducing Bacteria. C. J. P. Spruit and J. N. Wanklyn. (*Nature*, 1951, **168**, Dec. 1, 951–952). If bacteria aid corrosion by using up the hydrogen produced, the ratio N = (g. atoms iron corroded)/(molecules SO_4'' reduced) should equal 4 in the absence of organic hydrogen donors. In autotrophic media values ranging from 5 to 9 were obtained indicating that N is only a measure of the extent of hydrogen utilization by bacteria. The acceleration of corrosion in heterotrophic media by bacterial action is probably due to the production of sulphides.—A. G.

Dry Oxidation and Wet Corrosion. U. R. Evans. (*Nature*, 1951, **168**, Nov. 17, 853–855). An interpretation is attempted covering both high-temperature oxidation and wet corrosion. The electrochemical mechanism is discussed and the effects of inhibitors, sulphur, and impurities on corrosion are considered.

Corrosion-Proof Cements Extend Pickling Tank Life. C. F. Sauereisen. (*Iron Age*, 1951, **168**, Aug. 23, 66–68). The selection of the correct type of corrosion-proof cement for various applications is discussed. Silicate, sulphur, and resin cements are commonly used and an impervious membrane of rubber, lead, or a bitumastic composition is usual between the brick lining and outer shell.—A. M. F.

18-8 Can Be Cured after Intergranular Attacks. R. N. Gillmor. (*Iron Age*, 1951, **168**, Aug. 2, 81–85). Experiments have shown that those stainless steels which are susceptible to intergranular corrosion by carbide precipitation may be cured by holding them at the sensitizing temperature (750–1600° F.) for 1000 hr. or more. Chromium diffusion from the unaffected areas into the impoverished grain-boundary areas, rather than dissolving the precipitated chromium carbides, apparently effects the cure.—A. M. F.

Scaling of Gas-Turbine Alloys. A. Preece. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 149–169). The problem of high-temperature oxidation in the gas turbine is discussed, with special reference to the products of combustion of the fuel and impurities which may be present in the fuel.

Scaling of Heat-Resisting Steels: Influence of Combustible Sulphur and Oil-Fuel Ash Constituents. C. Sykes and H. T. Shirley. (Symposium on High-Temperature Steels and Alloys for Gas Turbines, 1951, *Iron Steel Inst. Special Report No.* 43, 153–169). For successful application in gas-turbine work heat-resisting steels must possess a very high resistance to scaling at the working temperatures. The problem is complicated by the ash and high sulphur contents of the heavy oil which is economically desirable as a fuel. The paper examines the effect of combustible sulphur and of potentially dangerous ash constituents, on the behaviour of a range of typical gas-turbine steels, under varying air/fuel ratios. Consideration is also given to the effect of molybdenum oxide, in view of the molybdenum content of some of the steels.

XUM

Tests on the Corrosion of Buried Iron and Steel Pipes. J. C. Hudson and G. P. Acock. (Symposium on the Corrosion of Buried Metals, Dec. 12, 1951, *Iron Steel Inst. Special Report No.* 45, 1–28). This report concerns the corrosion of cast-iron pipes and steel tubes buried in five representative soils in Great Britain for five years. The results relate to specimens tested both bare and with various protective coatings. For bare pipes, differences in the aggressiveness of the soil are of much greater moment than differences in the iron or steel. Because of their greater strength, centrifugally cast cast-iron pipes have a thinner wall than vertically cast cast-iron pipes of the same capacity. The latter should, therefore, last longer. In the present research, no marked difference in general rusting or pitting has been observed between the two types of cast iron. A zinc coating of 2 oz./sq. ft. protected the specimens over the five years, except in made-up ground consisting of ashes, where most of it had corroded away. A $\frac{1}{4}$-in. thick bitumen coating on steel tube was almost perfect at this site, but a thin hot-dipped coal-tar–pitch coating, 5 mils thick, on cast-iron pipe had failed. Protected specimens were removed from only one other site, in the Keuper Marl, where the bitumen coating was intact but the coal-tar–pitch coating showed signs of failure. Vitreous enamel on cast iron was in perfect condition at this site, but a stoved phenolic-resin coating on steel had failed because of its thinness.

Investigations on Underground Corrosion. K. R. Butlin, W. H. J. Vernon, and L. C. Whiskin. (Symposium on the Corrosion of Buried Metals, Dec. 12, 1951, *Iron Steel Inst. Special Report No.* 45, 29–38). Part I describes investigations on underground corrosion carried out at the Chemical Research Laboratory, Teddington, since 1934. Laboratory work includes fundamental studies on sulphate-reducing bacteria—their morphology, culture, and inhibition, and their effect on iron in anaerobic neutral conditions. Field investigations, mainly in collaboration with water and gas engineers, include direct observation of freshly exposed corroded pipes and the correlation of the presence of sulphate reducers with corrosion; tours of inspection of pipelines exposed at selected points; and (in collaboration with the Institution of Water Engineers) the collection of evidence from corrosion failures experienced by large water undertakings. It is concluded that the most widespread and dangerous form of underground corrosion takes place in neutral waterlogged soils and is associated with sulphate-reducing bacteria.

Part II describes experiments on the protection of ferrous pipes carried out by the Laboratory in collaboration with the Metropolitan Water Board. Tests with conventional protective materials (applied to an actual water system) suggest that adequate bitumen sheaths, concrete coverings, and gravel surrounds provide good protection. Other tests on unconventional protective materials (applied to buried test specimens) are in progress.

Corrosion of Buried Copper and Ferrous Strip in Natural and Salted Soils. British Electrical and Allied Industries Research Association. (Symposium on the Corrosion of Buried Metals, Dec. 12, 1951, *Iron Steel Inst. Special Report No.* 45, 39–54). An investigation to determine the relative corrodibility in different soils of various metals such as are, or might be, used for earth electrodes, and the relative corrosivity of the soils in relation to such metals, is described. The effect of salt treatment is discussed. and some comparison with other soil-corrosion studies is made. A general survey of the performance of various metals in the different soils is presented. No attempt is made to relate the corrosiveness of the soils to any characteristic other than the added salt.

Tests on the Corrosion of Buried Aluminium, Copper, and Lead. P. T. Gilbert and F. C. Porter. (Symposium on the Corrosion of Buried Metals, Dec. 12, 1951, *Iron Steel Inst. Special Report No.* 45, 55–74).

Cathodic Protection. K. A. Spencer. (Symposium on the Corrosion of Buried Metals, Dec. 12, 1951, *Iron Steel Inst., Special Report No.* 45, 75–94). The principal causes of corrosion of buried steel structures and the theory of protection by cathodic methods are briefly discussed before proceeding to the practical application of cathodic protection by both 'sacrificial' anodes and direct power supply. Survey methods and also the practical testing of cathodic protection systems are discussed. The financial saving reported for several existing cathodic protection installations, are quoted.

Cathodic Protection of Buried Metal Structures. R. de Brouwer. (Symposium on the Corrosion of Buried Metals, Dec. 12, 1951, *Iron Steel Inst. Special Report No.* 45, 95–110). Both theory and experiment show that buried metallic structures are protected against all types of corrosion when they are sufficiently electronegative with respect to their surroundings. Such cathodic protection may be obtained by polarized drainage, by the application of a current, or by the use of reactive anodes. Complementary arrangements may be provided to ensure a suitable distribution of current and to avoid all harmful action on neighbouring structures. Tests, involving electrode potential measurements, have been established to determine the best method of cathodic protection in a particular case, and to assess the efficiency of the method.

Study on Wear Phenomenon of Steel against Soil. 2nd Report. T. Mitsuhashi, Y. Imai, and S. Yokoi. (*J. Mech. Lab., Japan*, 1950, **4**, No. 8, 291–298). [In Japanese]. Results are given for experiments, using a testing apparatus previously described, on Arakawa and Soma sands. The relationships between amount of wear and velocity, cutting angle, load applied normal to the surface, and moisture content of the sand are indicated.—T. E. D.

External Ship Corrosion Due to Bacterial Action. W. S. Patterson. (*N.E. Coast Inst. Eng. Shipbuilders*, Dec. 7, 1951, Advance Copy). The author describes a very severe case of external ship corrosion. Bacterial action occurring in estuary mud is considered to have promoted the corrosion, aided by a period of unusually hot weather. The mechanism of the corrosion and means of preventing such attack are discussed.—R. A. R.

Corrosion Prevention in the Manufacturing Process. J. R. Fawcett. (*Mech. World*, 1951, **129**, Apr. 27, 381–384). A general survey is given of methods of protecting iron and steel from corrosion. Corrosion prevention can only be tackled effectively by serious consideration at the design stage.—D. H.

The Formation of Sigma and Its Influence on the Behaviour of Stabilized 18 per cent Chromium–8 per cent Nickel Steels in Concentrated Nitric Acid. R. S. Stewart and S. F. Urban. (*Symposium on the Nature, Occurrence, and Effects of Sigma Phase: Amer. Soc. Test. Mat.*, 1950, *Spec. Tech. Pub.* 110, 128–145). The presence of sigma phase is responsible for the low corrosion resistance of titanium-stabilized austenitic stainless steels in boiling 65% HNO_3, when tested after a 'sensitizing' treatment. Close control of the aluminium, chromium, and nickel contents reduced the formation of the sigma phase. A slight increase in the aluminium content increases the amount of delta ferrite to a moderate degree, but markedly increases the amount of sigma phase transformed from the ferrite present when the steel is suitably heated. As the annealing temperature is increased from 1800–2300° F. the amount of sigma formed on 'sensitizing' is increased, and corrosion resistance is decreased.—J. B. B.

ANALYSIS

Comparative Study of the Results of Spectrum Analysis and Chemical Analysis—Importance of the Standards. J. Gillis and M. J. Eeckhout. (*G.A.M.S. Colloque International de Spectrographic de Strasbourg*, Oct. 12–14, 1950, 31–55). The results of spectrochemical analysis are normally computed from a curve obtained by means of standard samples having compositions known by chemical analysis. To determine the accuracy of the spectrochemical results, comparisons are made with those obtained from chemical analysis. Results of investigations on loams, clays, and slags were compared with those obtained by several chemical laboratories. A large number of such comparisons showed that spectrochemical determinations of some components give better results than those obtained by chemical analysis in industrial laboratories. Sometimes it is not possible to plot a calibration curve with satisfactory precision because of the marked scatter of the results of chemical analyses carried out in different laboratories. It is therefore concluded that there is a lack of standard samples of accurately known composition. The creation of an Institute for the preparation of standard samples and for the analysis of samples, using precise and standardized methods, would be of great theoretical and practical value.

Carbons for Spectrography. Parisot and Badoz. (*G.A.M.S. Colloque International de Spectrographie de Strasbourg*, Oct. 12–14, 1950, 57–60). Commerical carbons, even the

'electrographites,' contain impurities in larger quantities than can be tolerated in analytical spectroscopy. The authors give the properties of their pure carbons, Nos. 207 and 208.

Convenient Method of Calculating in Spectrum Analysis by a System of Multiplying Factors. M. F. Hasler. (*G.A.M.S. Colloque International de Spectrographic de Strasbourg*, Oct. 12–14, 1950, 61–73). The author shows that with stainless and high-speed steels, when the basis metal content varies within wide limits it is necessary to employ a correction factor 'f_n (Cn)' to obtain the correct line-intensity ratios, but with low-alloy steels the effect of the basis metal concentration is negligible. The theory of the calculation of the correction factors is explained and examples are given showing the accuracy obtained with it using data determined with the Armament Research Laboratory Quantometer.

Analysis of Stainless Steels, Covering an Extensive Range of Contents, by Means of the Quantometer. M. F. Hasler. (*G.A.M.S. Colloque International de Spectrographic de Strasbourg*, Oct. 12–14, 1950, 75–79). A procedure for the spectrographic analysis of a wide range of stainless steels, using the Armament Research Laboratory Quantometer, is described. It is applicable to the following ranges: Fe 50·0–85·0, Cr 10·0–30·0, Ni 0·1–25·0, Mn 0·1–2·0, Si 0·1–1·0, Mo 0·1–2·5, Ti 0·1–2·0, Nb 0·1–1·0, and W 0·5–2·0%.

The Automatic "Spectro Reader" for Direct-Reading Spectrum Analyses. F.-C. Mathieu. (*G.A.M.S. Colloque International de Spectrographie de Strasbourg*, Oct. 12–14, 1950, 87–119). The equipment consists of: (1) A 'reading head' in place of the photographic plate, attached to a prism spectrograph of classical design. (2) An operating unit, mechanically connected to the 'reading head.' (3) An amplifier unit with oscilloscope and recording potentiometer. (4) A Faraday cage shielding and screening the source unit and the spark stand. The 'reading head' contains two photomultiplier tubes. One can be set on the basis metal (internal standard) while the other can be moved automatically (or manually) along the spectrum to the position of the line chosen for the analysis. During the adjustment to this line, a potentiometric measurement of the tube signals is recorded on a paper strip. The chief characteristic of the apparatus is that the periodicity of the sparks can be used to enhance the A.C. amplification of the photocell impulses.

The Search for the Cause of Calibration Line Drift in the Spectrographic Analysis of Steel. E. H.-S. Van Someren. (*G.A.M.S. Colloque Internattional de Spectrographie de Strasbourg*, Oct. 12–14, 1950, 121–125). The work of several laboratories seeking the cause of index point shift is summarized, most of the results being negative. The effect of humidity on the spectrum is traced to the photographic plate not the light source.

Display Microphotometry. F.-W.-J. Garton and R.-J. Webb. (*G.A.M.S. Colloque International de Spectrographie de Strasbourg*, Oct. 12–14, 1950, 127–143). This method of display microphotometry affords a very rapid and sensitive means of quantitative measurement for the underexposed portion of a spectrogram which has hitherto been of little or doubtful value to the spectrographer. It is more rapid in operation than normal microphotometry. Furthermore, the linear parameters of the standard curve obtained by this technique indicate many potential uses, such as in determination of limits of detection and of 'blanks.' The flexibility associated with cathode-ray tube presentation offers certain advantages over the more conventional pen-recording instruments.

Phenomena Resulting from Temperature Excitation in the Carbon Arc. W. Rollwagen. (*G.A.M.S. Colloque International de Spectrographic de Strasbourg*, Oct. 12–14, 1950, 145–147). Variable quantities of sodium introduced into a D.C. arc, together with constant quantities of other elements (of the order of 1 to 10 mg.) change the temperature of the arc. Every element having an ionization potential higher than that of sodium is excited to a maximum value at a definite concentration of sodium.

Relation between the Spectral Character of Spark Discharges and the Electrical Data. H. Kaiser. (*G.A.M.S. Colloque International de Spectrographie de Strasbourg*, Oct. 12–14, 1950, 149–154). The triggering of spark sources controls the electrical constants of the circuits. The question is then how to choose these constants in order to obtain conditions of vaporization and excitation suitable for analytical work. Sparks can be produced from a Feussner spark source (17,000 V.) or a Multisource (1000 V.) which are spectroscopically equivalent. Investigations have shown that the following two parameters completely determine the spectral character of the discharge: (1) the energy dissipated in a single train of oscillations, and (2) the duration of such a train.

Using a Hollow Cathode and a Vacuum Furnace in Spectrum Analysis. B. Rosen and M. I. Ottelet. (*G.A.M.S. Colloque International de Spectrographie de Strasbourg*, Oct. 12–14, 1950, 155–167). A new type of hollow cathode discharge tube specially designed for spectrographic studies is described. The hollow cathode is a thin-walled graphite crucible, in which the sample is placed, and it is heated to various high temperatures (2500° C.) by the discharge in permanent gas atmosphere. When the temperature increases, the lines of the various elements appear successively, the order of appearance being chiefly determined by the vapour pressure.

The Determinations of Halogen as well as Sulphur and Selenium by Spectrochemical Methods. P. A. Gatterer. (*G.A.M.S. Colloque International de Spectrographie de Strasbourg*, Oct. 12–14, 1950, 173–179). A simple and rapid method is described for the determination of the halogens, sulphur and selenium. A small quantity (10 to 20 mg.) of the sample is placed in a hard glass tube, which is evacuated to less than 0·001 mm. Hg., and the spectrum is excited by means of a high-frequency magnetic field.

Analysis of One Phase of an Alloy and Evidence of the Physicochemical State of an Element from Its Emission Spectrum. H. Triché. (*G.A.M.S. Colloque International de Spectrographie de Strasbourg*, Oct. 12–14, 1950, 185–190). A method for the spectrographic analysis of one phase in an alloy is described. Applications of this method to metallography include the determination of solid solubility and the distribution of impurities between two phases. Sources of error include: the electrochemical factor and the influence of the structure of the alloys and of the nature of the constituents on the spectral emission of the elements. The physicochemical state of an element is revealed by the vapour pressure of that element. With the excitation source used (a Tesla discharge) an increase in the vapour pressure results in an increase in intensity of lines due to the neutral atom as compared with those of the ionized atom.

The Organization of Spectroscopic Groups in Great Britain. D. M. Smith. (*G.A.M.S. Colloque International de Spectrographie de Strasbourg*, Oct. 12–14, 1950, 191–196). In 1927, the Brit. Non-Ferrous Met. Res. Assoc. had already established a Sub-Committee to deal with metallurgical applications of the spectrograph. Owing to the increasing interest shown in spectrographic methods and the analytical demands precipitated by the war, other Groups were formed, and the following British Groups exist today: The Spectrographic Discussion Group, Glasgow; The Physical Methods Group (S.P.A.); The Industrial Spectroscopy Group (Institute of Physics); The Photoelectric Spectrometry Group; The Spectrographic Discussion Group, Sheffield; and the Spectrographic Sub-Committee (Brit. Iron Steel Res. Assoc.).

The Position and Organization of Spectrochemistry in Germany. W. Seith. (*G.A.M.S. Colloque International de Spectrographie de Strasbourg*, Oct. 12–14, 1950, 197–200). The Central Committee for Spectrochemistry and Applied Spectroscopy in Germany is concerned with the development of spectrographic methods of analysis, primarily of metals and alloys. At the present time, however, it covers the whole field, including absorption, infra-red, and X-ray spectroscopy. This Committee became affiliated to the German Society of Metallurgy and it collaborates with other scientific commisionss.

The Italian Spectrochemical Research Centre. O. Masi. (*G.A.M.S. Colloque International de Spectrographie de Strasbourg*, Oct. 12–14, 1950, 201–202). Details of the organization and objects of this Centre are given.

The Swiss Association for Spectrographic Analysis. F. Dätwyler. (*G.A.M.S. Colloque International de Spectrographie de Strasbourg*, Oct. 12–14, 1950, 203). This Association was formed in 1938 and has now 44 members, 86% of whom are engaged in industrial, and 14% in scientific research. Two conferences are held each year.

The Direct-Reading Analysis of Boron in Low-Alloy Steels. (*Spectrographer's News Letter*, 1951, **4**, Sept.–Oct., 1–2). Details are given of the spectrochemical determination of 0·0005–0·02% boron in steels using the 2496·78 Å. boron

line. The ARL Production Control Quantometer is employed in conjunction with a flat sample used as anode, and a graphite rod counter-electrode.—T. E. D.

Furnaces for the Carbon and Sulphur Determinations in Pig Iron and Steel. F. H. Zeller. (*Vereinigte Österreichische Eisen- und Stahlwerke Jahrbuch* 1950–51, 137–141). Silicon carbide elements previously used in furnaces for the determination of iron and sulphur by the V.Ö.E.S.T. were replaced by Kanthal-wound furnaces operating at 1260–1280° C. The new furnaces were found to be considerably superior and more economical, having more than double the life of the old and being entirely satisfactory from the point of view of the accuracy of analytical results.—P. F.

Absorptiometric Determination of Chromium in Iron and Steel. (*J. Iron Steel Inst.*, 1952, **170**, Mar., 268–270). [This issue].

Macroscopic Estimation of Hydrogen in Solid Steel. E. V. Podol'skaya and E. G. Shumovskii. (Zavodskaya Laboratoriya, 1950, **16**, No. 2, 168–173). A method for determining hydrogen in steel, based on coating the specimen with enamel and observing the distribution, number, and size of blisters and craters, is described.

Autoradiographic Determination of the Lead Distribution in 18/8 Steel. L.-G. Erwall, A. Franzén, and M. Hillert. (*Jernkontorets Ann.*, 1951, **135**, No. 6, 219–228). [In Swedish]. The distribution of 0·004% lead in nickel–chromium steel was studied radiographically and an enrichment of lead was detected between the dendrites first formed on solidification.—B. S. E.

Rapid Photometric Determination of Lead in Iron Ores. F. Neuwirth. (*Vereinigte Österreichische Eisen- und Stahlwerke Jahrbuch* 1950–51, 133–136). Lead in iron ores is transformed into the red lead-diphenylthiocarbazon which, in solution in carbon tetrachloride, is photometrically titrated by means of the extinction method with a Pulfrich photometer. Pure carbon tetrachloride is used as blank. Very close agreement between photometric and gravimetric results with the ores tested, which contained from 0·03% to 0·76% of lead, was obtained. Details of the analytic procedure are given.—P. F.

Recommended Methods for the Sampling and Determination of Moisture in Coke, at Coke Ovens and Blast Furnaces. (*Joint Coke Consultative Committee*, June, 1950). In this report of the South Yorkshire Coke Quality Panel methods for use by both producer and consumer in determining moisture are reported. The Panel concludes that most of the values obtained in the past were liable to be in error by at least 100%. It is commercially impracticable to determine accurately the moisture content in a single wagon of coke. The only satisfactory method is to determine the mean moisture in coke supplied from a particular source over a period. The use of quality control charts at the coke ovens is recommended, using shift sub-sample figures to indicate and control the variability of the coke during the period concerned.

Determination of Nitrogen in Coal. G. N. Badami and J. W. Whitaker. (*Fuel*, 1951, **30**, Sept., 211–212). In the Kjeldahl determination manganese dioxide or potassium permanganate may be employed with advantage. When using permanganate, the coal (0·1 g.) is treated with boiling acid (10 ml.) for 5 min. before any permanganate is added. An after-boil of 20 min. is adequate.—J. P. S.

ECONOMICS AND STATISTICS

Saving Scarce Materials. (Anglo-American Council of Productivity, Nov., 1951). This 28-page report covers the work of a team of eight which spent 26 days in the U.S.A. during which 53 conferences were held and many visits to plants were made. It is recommended that existing steel conservation committees in the United Kingdom should give renewed attention to the properties of low-alloy steels and to special heat-treatment techniques.—R. A. R.

Newcastle, Port Kembla and Whyalla, Australia. (*Usco Magazine*, 1951, **2**, June, 11–13). A short description is given of the towns Newcastle, Port Kembla, and Whyalla, Australia, with particular attention to the iron and steel industries centred on them.—D. H.

World Iron Production in the Year 1949. R. Durrer. (*Von Roll Werkzeitung*, 1950, **21**, Nov., 225–227). Comparison of the year 1949 with 1948 shows a drop of 7 million tons in pig iron production to 51·5 million tons, and of steel ingot production from 83·3 to 73·6 million tons in North America. Elsewhere, however, there were rises in the production of pig iron and steel. Data for Western Europe and for Russia and her allies are given. Were it not for the demand created by rearmament the capacity of the industry would be too large.

MISCELLANEOUS

Supply and Use of Hydraulic Power at Appleby-Frodingham. L. N. Lloyd. (*J. Iron Steel Inst.*, 1952, **170**, Mar., 271–287). [This issue)]

Dust and Dust Separators in the Iron and Metal Industries. B. Nyrud. (*Jernindustri*, 1951, **32**, Feb., 28–34; Mar., 42–45; May, 73–74). [In Norwegian]. The author stresses the hygienic reasons for abatement of air-borne dust in the physiologically active range (5–0·2 μ). The Swedish Biéth cyclone and the Linderoth dynamic air filter are described.

Architecture in the Planning of Modern Industry. F. Reischl. (*Vereinigte Österreichische Eisen- und Stahlwerke Jahrbuch* 1950–51, 160–164). The achievement of aesthetic harmony through the choice of definite dimensional relationships between different parts of a structure and between different neighbouring structures of industrial units is discussed. Historical and modern methods of fixing dimensions in relations pleasing to the eye are considered, and their application in the design of rolling mills for V.Ö.E.S.T. is considered and illustrated by drawings and photographs.—P. F.

BOOK NOTICES

ANDREWS, P. W. S., and ELIZABETH BRUNNER. "*Capital Development in Steel.*" A Study of The United Steel Companies, Ltd. La. 8vo, pp. x + 374. Illustrated. Oxford, 1951 : Basil Blackwell. (Price 30*s.*)

The publication of this book is most opportune, because although it is only intended to provide a study of capital development or investment in The United Steel Companies Limited, it is virtually a history of the heavy steel industry during the last hundred years. The authors exhibit no political bias and anyone who wishes to form his own views concerning the desirability or otherwise of nationalizing the British iron and steel industry should, in the first place, read this most instructive work. The general reader who is chiefly aware of steel as a political battleground and is familiar with the accusations levelled at the steelmasters, will find the answers to such questions as—Was output restricted to keep prices up? Was the industry inefficient? Was the capital equipment allowed to run down? etc.

The two authors are engaged on a programme of industrial research at Nuffield College and obtained permission from The United Steel Companies Limited to make the enquiry which resulted in the present book, and all readers will readily acknowledge the generosity of the Chairman and Board of Directors in giving them freedom to publish details which are usually locked away in secret archives. The only restrictions that were imposed were those which were obviously necessary when a public account was to be presented of a business which is still very much alive, and they stem from a consideration that whilst its past history may be open to the public, its future policy is still a private matter. Throughout the book the authors have allowed for the interest that both non-economists and non-steelmen may be expected to have. Thus, economists and technicians will on occasion find explanations, etc., which would be unnecessary if the book were intended for a narrow professional audience of one or the other.

After a general introductory chapter the authors devote a chapter to techniques and processes in order that the reader may have a proper appreciation of the manufactures carried on by the different firms broadly classified as steel manufacturers. Sections are devoted to the blast-furnace, and steelmaking by the Bessemer and by the open-hearth processes, by electric furnaces, and by the various duplex processes. The chapter concludes with sections describing ingots and their forging and rolling, and the further treatment of hot-rolled product.

In chapter 3 the authors outline the history of the British steel industry between the wars, and describe The United Steel Companies as it is at the present time. The fourth chapter describes the development of the Companies down to 1927 and begins with the formation of the combine and the heavy capital investment which followed and its (the combine's) reaction to the storms of the 1920's.

Chapter 5 describes the managerial reorganization and the 'rationalization' of the combine between 1928 and 1930, and the sixth chapter studies the progress of the Companies from 1930 down to the outbreak of war. Capital development since the war is covered by chapter eight, in which a table giving the estimated earnings or savings on individual projects is of particular interest, whilst chapter nine describes the capital development that was actually carried out, and takes the history of the Companies to June 1950, when it began another extensive stocktaking of its plans and policies.

Chapter ten relates the findings of the research.

This is an extremely well written book and, although it deals essentially with the steel industry, all readers will be interested in the references to the problems and dangers generally inherent in public ownership.

J. FERDINAND KAYSER

BRITISH CAST IRON RESEARCH ASSOCIATION. "*Typical Microstructures of Cast Iron.*" 4to, pp. 55. Illustrated. Alvechurch, Birmingham, 1951: The Association. (Price 21*s.*)

A number of collections of illustrations of the structures of metals and alloys as viewed under the microscope have been published; one of the best known was the comprehensive collection published in Berlin before the last war by H. Hanemann and A. Schrader under the title "Atlas Metallographicus," whilst others, less comprehensive but more specialized, were issued both in America and in Great Britain. In 1933 and 1936, the British Cast Iron Research Association collaborated with the Institute of British Foundrymen in preparing two short booklets illustrating some microstructures of cast iron for the use of foundrymen. The present work is intended to provide for reference and comparison a series of photomicrographs of the principal types of cast iron normally encountered by the metallurgist and engineer in their daily work, at magnifications to which they are accustomed; from these they will be able to estimate how far a given structure is likely to meet specified requirements. The book is provided with a technical introduction which indicates the dependence of the structure and properties of cast iron on the cooling rate and composition, and considers separately the mode of occurrence and appearance of the constituents of cast iron. The photomicrographs, which are 41 in number, are printed on large-size paper (11 × 8½ in.) and are accompanied in each case by an explanatory text on the facing page. They are grouped under constituents of cast iron, unalloyed cast irons, special and alloy cast irons, and malleable cast irons, and the work concludes with a few photomicrographs illustrating some of the commoner defects encountered in iron castings. The microphotographs are of the highest quality and their reproduction is excellent. The book is in every way very well got up.—A. E. C.

ERDMANN-JESNITZER, F. "*Werkstoff und Schweissung. I.*" 8vo, pp. xxiv + 1002. Illustrated. Berlin, 1951: Akademie-Verlag. (Price DM 160.– for 2 vols.)

The compilation of encyclopaedic textbooks was one of the great achievements of pre-war German scholarship. With diligence and erudition a subject was pursued from its elements into all its nooks and crannies, until no avenue had remained unexplored, no stone unturned, and all connections and ramifications were uncovered. Unwieldy these tomes sometimes were, but one could always be sure that if information could not be found in the book it did not exist. For comprehensiveness, thoroughness, and scientific rigour, the German textbook had few competitors.

A welcome rebirth of this spirit is apparent in this two-volume work on welding technology, of which the first volume, under review, is devoted to ferrous materials and the second volume, in preparation, will deal with non-ferrous materials.

In the preparation of this monumental work, the editor appears to have invited his authors to fill in a rough skeleton, so producing what in Great Britain is called a symposium. The inevitable result is a certain amount of repetition and overlap, and of course there are gaps, but the advantage of giving authors a fairly free hand is worth this sacrifice.

The present volume, of over a thousand pages, is divided into 39 contributions by 32 authors which are grouped under the four headings: Manufacture and Weldability; Processes; Special Welding Problems; and Design and Calculations.

Only two contributions are offered under the last of these four main headings, one of less than ten pages on the important subject of Chemical Vessels by W. Küntscher, and the other of just under a hundred pages by A. Erker and A. Thum on "*Gestaltfestigkeit.*" This word, which cannot be translated, was introduced by Thum to convey the idea that the strength of a structure is not directly related to the strength values of the materials determined in conventional tests but depends entirely on the form of the structure and its details. Whilst Thum's contribution is most interesting and highly original, it is scarcely adequate to cover the design of welded structures. One can only hope that perhaps room will be found in the second volume to cover other aspects of the design problem, and that more room will be found also for welded pressure vessels and pipelines than the ten pages devoted to this subject in the present volume.

Whereas roughly only 100 pages are devoted to design, 22 of the contributions—that is, well over half the book—are concerned with welding processes and their technicalities, and one may feel that thoroughness in this respect has perhaps gone too far at the expense of other equally or more important aspects. One notices the complete absence of a section on low-temperature notch brittleness of steels. Brittle fracture is only discussed quite briefly as if it were part of the weldability problem. Perhaps this must be excused in view of the fact that Germany has been cut off for many years from all sources of foreign information, and, whilst the problem first arose on the Continent in the failure of German and Belgian bridges, all the work advancing this problem towards a solution has been carried out in Great Britain and the United States. It is to be hoped that in any future editions this serious shortcoming will be remedied.

There are two extremely interesting contributions which one would normally not expect to find in a book on welding technology. One, by H. Hagen, is a dimensionless analysis of heat-flow problems, and important relations are established which permit the study of the thermodynamic aspects of welding from a new angle with the aid of models. Whether the main application of these new relations will be found in the study of distortion and residual stresses, as the author seems to think, is doubtful, because the fundamental problem is not to determine the stresses in a given structure but to ascertain their effect, and this cannot be predicted from merely knowing their magnitude and distribution. Nevertheless Hagen's contribution may well be the starting point to a more thorough understanding of many welding problems. The other contribution, by W. Rätalaer and W. Dawihl, is devoted to a discussion of certain relations which have been found between wear and abrasion and welding.

It is clearly impossible to give within this review a translation of the titles of all the 39 contributions, let alone a considered criticism, but one other contribution shall be mentioned, and this is K. L. Zeyen's article on the development of electrodes for fusion welding. No publication in English is known to the reviewer to match this excellent, thorough, and comprehensive discussion of the mysteries of electrode manufacture and coating compositions.

For all those more deeply concerned with welding this book should prove an invaluable and indispensable source of information, and it is to be hoped that some enterprising British or American publisher will translate it into English.

R. WECK

"*Gmelins Handbuch der anorganischen Chemie.*" Achte völlig neubearbeitete Auflage "Titan." Mit 100 Figuren. System-Nummer 41. La. 8vo, pp. xxii + 481. 1951: Verlag Chemie G.m.b.H., Weinheim/Bergstrasse. (Price DM 113.–)

For anyone wishing to find the sources of information on the occurrence of titanium ore, preparation of the metal,

and properties of titanium, its alloys, and compounds, this, the latest of the Gmelins Handbuch series, contains without doubt the most comprehensive collection of references, classified and indexed with the thoroughness which one has come to expect with each fresh issue in the series. It must be borne in mind, however, that publications after December, 1949, are not included.

Of the present book, one-sixth deals with the occurrence of titanium-bearing ores, one-tenth with methods of producing the metal, one-seventh with the properties of titanium itself, and the remainder, some 280 pages, with the properties of its alloys and compounds.

The ferrous metallurgist will look in vain in the index for any reference to titanium–iron alloys or the titanium–carbon–iron system, which is rather surprising as L. Northcott in his summary of published information on titanium and its effects upon iron and steel (*Iron Steel Inst. Special Report No.* 24, 1939) refers to Lamort's melting-point diagram of iron–titanium alloys, and Hansen's review of this system. The only references the reviewer can find on titanium in relation to iron and steel are three or four (p. 358) in the section on the titanium–carbon system.

R. A. R.

OLSEN, GERNER A. "*Strength of Materials.*" 8vo, pp. vi + 442. Illustrated. London, 1951 : George Allen and Unwin, Ltd. (Price 32*s.* 6*d.*)

The author of this book is Assistant Professor of Civil Engineering at the City College of New York. He states that it is designed for use in technical institutes and colleges offering courses in strength of materials where calculus is not required. The majority of British students for whom the book would be suited do study calculus, so that treatment of the subject without directly invoking calculus is of no particular advantage to such readers. Of course, the author himself has to use calculus, but in this book only the results are stated.

As far as can be seen the author has not attempted to modify the book for the English market. Thus, whilst the occasional use of different symbols or the working of problems in pounds instead of tons are of no real account, the fact that only American codes are referred to in connection with riveting and welding, for example, does detract somewhat from the value of the book in Great Britain. Surely this point could have been covered comparatively easily, if only by footnotes?

It is a conventional textbook ; it is considered to be suitable for Higher National Certificate and largely for Higher National Diploma students in engineering, and would also serve as a useful introduction for a degree student, but the treatment seems to be too wide for a metallurgical student studying the strength of materials.

The book is clearly written and well produced. The illustrations are excellent and there are numerous worked problems. There is no doubt that it makes easily understood reading.—A. R. BAILEY

NEW PUBLICATIONS

BOUSSARD, FRANÇOIS. "*Défauts de Fonderie.*" Liège : Georges Thone.

BROWN, CHARLES L. "*Basic Thermodynamics.*" 8vo, pp. x + 266. Illustrated. New York, 1951 : McGraw-Hill Book Co., Inc. (Price $4.50)

DARCY, M. "*Pour le forgeron.*" Vol. I, 3rd edition : 8vo, pp. vi + 158. Illustrated. Vol. II, 3rd edition : pp. vi + 142. Illustrated. Paris, 1951 : Dunod. (Price 290 fr. per vol.)

DOE, E. W. "*Foundry Work.*" Large 8vo, pp. 109. Illustrated. New York, 1951 : John Wiley and Sons, Inc. (Price $1.76)

EVANS, U. R. "*Précis de corrosion.*" Une introduction à l'étude des réactions entre corps métalliques et non-métalliques. Translated by G. Dechaux. Large 8vo, pp. xxii + 258. Illustrated. Paris, 1951 : Dunod. (Price 1750 fr.)

FROCHT, M. M. "*Strength of Materials.*" La. 8vo, pp. 439. New York, 1951 : Ronald Press Company. (Price $5.50)

GILL, J. P., G. A. ROBERTS, H. G. JOHNSTIN, and GEORGE BURNS. "*Aciers d'outillage.*" Translated by Michel Barba. 8vo, pp. xvi + 551. Illustrated. Paris, 1951 : Dunod. (Price 2850 fr.)

GIRSCHIG, ROBERT. "*La Métallurgie des Poudres.*" Préface de l'ingenieur général Nicolau. 8vo, pp. 100. Illustrated. Paris : Editions de la Revue d'Optique.

"*Gmelins Handbuch der anorganischen Chemie.*" Achte Auflage. System-Nummer 3. "*Sauerstoff.*" Lieferung 2, "*Vorkommen. Technologie.*" 8vo, pp. iv + 83–300. 1952 : Verlag Chemie, G.m.b.H., Weinheim/Bergstrasse. (Price DM 65.–)

KAESSBERG, HUGO. "*Gesenkschmieden von Stahl.*" Band 2. "*Die Gestaltung der Schmiedewerkzeuge.*" 2. neubearb. Aufl. 8vo, pp. 62. Illustrated. (Werkstattbücher für Betriebsangestellte, Konstrukteure und Facharbeiter. H.58.). Berlin, 1951 : Springer-Verlag. (Price DM 3.60)

LEWIS, BERNARD, and GUENTHER VON ELBE. "*Combustion, Flames, and Explosions of Gases.*" Pp. xix + 795. Illustrated. New York, 1951 : Academic Press, Inc. (Price $13.50)

MAYER-SIDD, EUGEN, and FRANTZ HUTTERER. "*Merkbuch für Fehler bei der Warmbearbeitung von Eisen und Stahl.*" Fehler des Stahles, Stahlauswahl, Schmieden, Glühen, Härten, Anlassen, Vergüten, Nieten, usw. 5. Aufl. 8vo, pp. 131. Illustrated. Stuttgart, 1950 : Berliner Union. (Price DM 4.60)

METAL CUTTING TOOL INSTITUTE. "*Machining of Stainless Steels.*" Pp. 27. New York : The Institute. (Price $1)

MEYENBERG, F. L. "*Industrial Administration and Management.*" 8vo, pp. 387. London : Sir Isaac Pitman and Sons, Ltd. (Price 35*s.*)

MICHELL, A. G. M. "*Lubrication.*" London and Glasgow : Blackie and Son, Ltd. (Price 35*s.*)

MURNAGHAN, FRANCIS D. "*Finite Deformation of an Elastic Solid.*" 8vo, pp. 140. New York, 1951 : John Wiley and Sons, Inc. (Price $4)

"*Phase Transformations in Solids.*" Edited by a committee composed of R. Smoluchowski, chairman, J. E. Mayer, and W. A. Weyl. Pp. x + 660. New York, 1951 : John Wiley and Sons, Inc. (Price $9.50)

PRAGER, WILLIAM, and PHILIP G. HODGE, jun. "*Theory of Perfectly Plastic Solids.*" 8vo, pp. 264. Illustrated. New York, 1951 : John Wiley and Sons, Inc. (Price $5.50)

SCHLESINGER, G. "*Messung der Oberflächengüte.*" Pp. 248. Illustrated. Berlin, 1951 : Springer-Verlag. (Price DM 31.50)

SOCIÉTÉ BELGE DES MÉCANICIENS. "*Code de Bonne Pratique pour la Realisation des Modèles de Fonderie.*" 4to, pp. 20. Brussels, 1951 : The Society. (Price 50 fr.)

STEEL FOUNDERS' SOCIETY OF AMERICA. "*Recommended Practice for the Welding of Steel Castings.*" Cleveland, Ohio, 1951 : The Society. (Price 35 cents)

STEINER, K. "*Fortschritte in der Prüfung von Rost- und Wetterschutzanstrichen ; Neuerungen auf dem Gebiet der Rostschutz-Pigmente : Moderne Bindemittel für Rost- und Wetterschutzfarben.*" La. 8vo, 3 vols., pp. 18, 20, 26. Garmisch-Partenkirchen, 1951 : Fachliteratur Ermittlungs- und Berichtdienst. (Prices DM 10.50, 12.–, and 15.– respectively)

"*The Wire Reference Year Book and Directory.*" Festival of Britain Edition. Oswestry (Salop), 1951 : Owen's Library Printing Works. (Price 30*s.*)

TYVAERT, P. "*Protection contre la corrosion des surfaces métalliques.*" La. 8vo, pp. 88. Illustrated. Paris, 1951 : Dunod. (Price 440 fr.)

VOGEL, ARTHUR I. "*A Text-Book of Quantitative Inorganic Analysis. Theory and Practice.*" 2nd Edition. 8vo, pp. xxiv + 918. Illustrated. London and New York, 1951 : Longmans, Green and Co. (Price 48*s.*)

WAGNER, C. "*Thermodynamics of Alloys.*" 8vo, pp. 160. Illustrated. Massachusetts, 1952 : Addison-Wesley Press Inc. (Price $6.50)

WHITTAKER, A. "*Electroplating and the Engineer.*" 12mo, pp. 87. Manchester, 1951 : Emmott and Co., Ltd. (Price 4*s.*)

WILSON, WILBUR M., R. A. HECHTMAN, and W. H. BRUCKNER. "*Cleavage Fractures of Ships Plates.*" Pp. 95. Urbana, Ill., U.S.A. : University of Illinois. (Price $1)